



**DATA VALIDATION**

EPA Region 5 Records Ctr.



362890

**FOR**

**EAGLE ZINC PROJECT**

**INORGANICS ANALYSIS DATA**

**Target Analyte List Metals in Soil Samples**

**Samples Collected March 16, 2005**

**Chemical Analyses Performed by:**

**EnChem (Division of Pace Analytical Services, Inc.)**

**Green Bay, Wisconsin (All Analytes except Mercury)**

**Kimberly, Wisconsin (Mercury)**

**Sample Delivery Group Number 857185**

**Data Deliverables Prepared for:**

**ENVIRON International Corporation**

**Deerfield, Illinois**

**Data Validation Performed by:**

**Trillium, Inc.**

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**April 5, 2005**

05906/ESC/PAC  
857185 Metals

## EXECUTIVE SUMMARY

Validation of the inorganics analysis data [target analyte list (TAL) metals] was completed by Trillium, Inc. Analyses for mercury were performed by EnChem Laboratories in Kimberly, Wisconsin. Analyses for the remaining target analytes were performed by EnChem Laboratories in Green Bay, Wisconsin. The samples in this data set represent the sample collections from March 16, 2005, from the Eagle Zinc Project. Data for six soil samples were reported by the two laboratories for a single data package 2005r1 Sample Data Package Group (SD6) h [1]

following samples:

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A2-3-S1	A2-3-S1D	A2-13-S1	A1-26-S1	A1-3-S1	A1-3-S1-2
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Findings of the validation effort resulted in the following qualifications of sample results:

- Results for antimony, cadmium, and nickel in all of the samples in this data set were qualified as estimated (UJ, J).
- Results for zinc in all of the samples in this data set were qualified as estimated (J).
- Results for aluminum, copper, chromium, magnesium, and potassium in all of the soil samples in this data set were qualified as estimated (J).
- Results for silver in A2-3-S1, A2-3-S1D, and A1-3-S1-2, for selenium in A2-3-S1D, A2-13-S1, A1-26-S1, A1-3-S1, and A1-3-S1-2, and for thallium in A2-13-S1 and A1-3-S1-2 were qualified as estimated (J).

Brief explanations of the reasons for the actions taken above may be found in the Overall Assessment (Section XII). Details of the validation findings and conclusions based on the review of the results for each quality control requirement are provided in the remaining sections of this report.

The validator removed all “A,” “B,” “C,” “E,” “N,” and “\*” qualifiers applied by the laboratory.

Chain of custody documentation and data presentation issues are discussed in Section XI.

This validation report should be considered part of the data package for all future distributions of the inorganics data.



## INTRODUCTION

Analyses were performed according to EPA Methods 3050B, 7471A, 6010B, and 6020 (SW-846; Test Methods for Evaluating Solid Waste, Third Edition, through Update III, 12/96). Results of sample analyses are reported by the laboratory as either qualified or unqualified; various qualifier codes are used by the laboratory to denote specific information regarding the analytical results. In this data set, the laboratory used the following defined qualifiers:

- A Analyte is detected in the method blank. Method blank criteria are evaluated to the laboratory method detection limit. Additionally, method blank acceptance may be based on project specific criteria or determined from analyte concentrations in the sample and are evaluated on a sample by sample basis.
- B The analyte has been detected between the method detection limit and the reporting limit.
- C Elevated detection limit.
- E Estimated concentration due to matrix interferences. During the metals analysis the serial dilution failed to meet the established control limits of 0-10%. The sample concentration is greater than 50 times the IDL for analysis done on the ICP or 100 times the IDL for analysis done on the ICP-MS. The result was flagged with the E qualifier to indicate that a physical interference was observed.
- N Spiked sample recovery not within control limits.
- \* Precision not within control limits.

The validator removed all "A," "B," "C," "E," "N," and "\*" qualifiers applied by the laboratory.

To the extent applicable, Trillium's validation was performed in conformance with the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (EPA 540-R-01-008, July 2002). Where discrepancies were found, the specifications of the referenced methods took precedence. In addition, professional judgment was applied as necessary and appropriate.

The data validation process is intended to evaluate data on a technical basis rather than a contract compliance basis. This requires that the data package contain sufficient raw data documentation to facilitate the validation process and allow verification of all reported results. It is also assumed that the data package represents the best efforts of the laboratory and has already been subjected to adequate and sufficient quality review prior to submission for validation.



During the validation process, laboratory data are verified against all available supporting documentation. Based on this evaluation, qualifier codes may be added, deleted, or modified by the data validator. Validated results are, therefore, either qualified or unqualified. Unqualified results mean that the reported values may be used without reservation. Final validated results are annotated with the following codes as defined in the National Functional Guidelines:

- U      The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
- J      The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
- J+    The result is an estimated quantity, but the result may be biased high.
- J-    The result is an estimated quantity, but the result may be biased low.
- R      The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.
- UJ    The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

These codes are recorded on the Data Summary Form contained in Attachment A, as well as on the laboratory answer forms in Attachment B of this validation report to qualify the results as appropriate according to the review of the data package.

Two facts should be noted by all data users. First, **the “R” qualifier means that the laboratory-reported value is unusable.** In other words, due to significant quality control problems, the analysis is invalid and provides no information as to whether the compound is present or not. Rejected values should not appear on data tables because they cannot be relied upon, even as a last resort. Second, **no analyte concentration is guaranteed to be accurate even if all associated quality control is acceptable.** Strict quality control conformance serves only to increase confidence in reported results; any analytical result will always contain some error.

The data user is also cautioned that the validation effort is based on the raw data printouts as provided by the laboratory. Software manipulation cannot be routinely detected during validation; unless otherwise stated in the report, these kinds of issues are outside the scope of this review.

## **I. Holding Times, Preservation and Sample Integrity**

The soil samples were collected on March 16, 2005. All of the samples were digested and analyzed well within acceptable holding times (28 days for mercury and 180 days for the remaining analytes).

The temperature of the sample cooler (4°C), as recorded on the Chain of Custody Record and the Cooler Receipt Log, was within the acceptable temperature range (4°C  $\pm$  2°C).

The Chain of Custody Record and Cooler Receipt Log also indicated that the samples were received intact but no custody seals were used on the sample cooler.

Copies of the FedEx airbills were provided by the laboratory on April 4, 2005, to document custody of the samples between the field and the laboratory.

## **II. Mass Spectrometer Tune**

The tuning solution for the ICP-MS was analyzed on March 19, 2005 at 16:18. All criteria specified in Method 6020 were met.

As required by the analytical method, a blank and three calibration standards were analyzed to document instrument stability. The laboratory confirmed that three integrations of each target analyte were performed but only the average of these integrations was reported in the supporting data. Percent relative standard deviation (%RSD) values were reported by the laboratory but could not be confirmed by the validator since abbreviated raw data were provided. No acceptance limits are provided in Method 6020 but all reported %RSD values were less than 10%, which was deemed acceptable based on professional judgment.

## **III. Calibrations**

Inductively coupled plasma-mass spectrometry (ICP-MS) analyses were performed on an instrument identified as "HP ICPMS 2." The samples reported in this data set were analyzed in two analysis series on March 19 and 21, 2005.

Inductively coupled plasma-atomic emission spectroscopy (ICP-AES) analyses were performed on a trace ICP-AES instrument identified as "TJA61E Trace ICP 3." The samples reported in this data set were analyzed in a single analysis series on March 22, 2005.

Mercury analyses were performed in a single analysis series run on March 23, 2005 on a cold vapor atomic absorption (CVAA) system identified as "PE CVAA 1."



Initial calibration verification (ICV) standards were performed for all of the target analytes after each initial calibration. All ICV percent recoveries (%R) were correctly calculated, accurately reported, and within the validation guideline-specified control limits (90-110% for analytes determined by ICP-MS and ICP-AES and 80-120% for mercury).

Continuing calibration verification (CCV) standards were run at the appropriate frequency (after every ten samples) in all of the analysis series. All %Rs were correctly calculated, accurately reported, and within the validation guideline-specified control limits (90-110% for analytes determined by ICP-MS and ICP-AES and 80-120% for mercury).

A linearity check for mercury was performed at the start of the analysis series and gave an acceptable correlation coefficient ( $>0.995$ ).

The analysis of "CRI" standards in the ICP-MS and ICP-AES analyses and "CRA" standards in the mercury analyses are not required by SW-846 methods and were not performed in association with the reported analyses.

#### **IV. Blanks**

Initial and continuing calibration blanks (ICBs and CCBs) were analyzed at the appropriate frequencies throughout each analytical sequence. No target analytes were detected in any of the ICBs or CCBs.

Aluminum (11.925 mg/kg), calcium (28.545 mg/kg), chromium (0.175 mg/kg), copper (0.080 mg/kg), iron (29.319 mg/kg), lead (0.195 mg/kg), magnesium (8.855 mg/kg), manganese (0.130 mg/kg), potassium (10.010 mg/kg), sodium (21.845 mg/kg), zinc (0.832 mg/kg), and mercury (0.002 mg/kg) were detected in the solid-matrix preparation blank (PB). With regard to PBs, the validation guidance document indicates that if an analyte is detected in the PB below the contract required quantitation limit (CRQL), "no correction of the sample results should be performed." For these samples, the estimated quantitation limit (EQL) was substituted for the CRQL. *None of these results were detected at concentrations greater than the analyte-specific EQL; therefore, no data were qualified on this basis.* In addition, results for these analytes in the site samples were significantly greater than the concentrations detected in the PB (i.e., all were greater than five times the concentration detected in the PB).

No field-submitted blanks were included in this data set.

#### **V. ICP Interference Check Sample**

ICP interference check sample analyses were performed at the appropriate frequency throughout all analysis series. Target analyte recoveries were acceptable (80-120%) in all cases.

## VI Spiked Sample Analyses

### A. Laboratory Control Samples

A solid-matrix laboratory control sample pair (LCS/LCSD) was prepared and analyzed with each analysis series. Each LCS/LCSD pair included all of the target analytes required for that analytical sequence. All percent recovery (%R) and relative percent difference (RPD) values were correctly calculated, accurately reported, and within the analyte-specific acceptance limits specified by the laboratory on the summary forms.

### B. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analyses

A1-26-S1 was prepared as an MS/MSD pair for all of the target analytes. All %R and RPD values were correctly calculated and accurately reported. For the target analytes with an amount of spike added greater than four times the concentration of the analyte in the original analysis of A1-26-S1, %Rs and RPDs were within the analyte-specific acceptance limits specified by the laboratory on the summary forms with the following exceptions:

Analyte	MS	MSD	Acceptance Limits
antimony	51%	55%	80-120%
cadmium	136%		75-125%
nickel	125%	116%	75-110%

Based on unacceptably low recoveries in both of the matrix spike analyses, results for antimony in all of the samples in this data set were qualified as estimated (UJ, J). Based on unacceptably high recoveries in one or both of the matrix spike analyses and positive detection in the associated samples, results for cadmium and nickel in all of the samples in this data set were qualified as estimated (J).

For the target analytes with an amount of spike added less than four times the concentration of the analyte detected in the original analysis of A1-26-S1, acceptable recoveries would not be expected. Since the amount of spike added is insignificant, this presents an opportunity to compare precision among the triplicate analyses (sample, MS, and MSD). Based on professional judgment, an acceptance limit of  $\leq 25\%$  RSD was used to evaluate the precision among results reported for aluminum, barium, copper, iron, lead, manganese, and zinc. With the exception of zinc (26.1%), acceptable precision among the triplicate results was observed (2.3% to 16.3%). Based on poor precision in the triplicate analyses, results for zinc in all of the samples in this data set were qualified as estimated (J).

Post digestion spike recoveries for all of the TAL target analytes in A1-26-S1 were acceptable (75-125% acceptance limits).

## **VII. Duplicate Sample Analyses**

### **A. Laboratory Duplicate Analysis**

No laboratory duplicate analysis was performed on a sample in this data set.

### **B. Field Duplicate Analyses**

No field duplicate pair was identified in this sample set.

## **VIII Internal Standard Performance**

Lithium (mass 6), scandium (mass 45), germanium (mass 74), yttrium (mass 89), indium (mass 115), terbium (mass 159), and bismuth (mass 209) were included as internal standard analytes for the ICP-MS analyses. Percent recoveries of the internal standards were within the analytical method-specified 30-120% acceptance limits in all of the site and quality control sample analyses.

## **IX ICP Serial Dilution**

ICP-AES and ICP-MS serial dilution analyses were performed on A2-3-S1. Results for elements with initial (undiluted) results greater than 10 times the instrument detection limit for analytes determined by ICP-AES and greater than 100 times the concentration of the analyte in the reagent blank for analytes determined by ICP-MS were acceptable (<10% Difference) with the exceptions of aluminum (11%), copper (12%), chromium (12%), magnesium (17%), nickel (25%), and potassium (14%). Based on unacceptable results in the serial dilution analyses, results for aluminum, copper, chromium, magnesium, nickel, and potassium in all of the soil samples in this data set were qualified as estimated (J).

## **X. Sample Results Verification**

For the ICP-MS analyses, the laboratory confirmed that three integrations of each target analyte were performed but only the average of these integrations was reported in the supporting data; therefore, the reported average concentration could not be confirmed by the validator. Based on the reported average concentrations for the analytes determined by ICP-MS and for all remaining target analytes, all sample results were correctly calculated and accurately reported from the raw data, including adjustments for the solid-matrix preparation procedures, dilutions, and percent solids.





Instrument detection limits (IDLs) were reported for all three instruments used to perform the reported sample results. IDLs were established on April 4, 2004 on the ICP-MS instrument, on January 5, 2004, on the ICP-AES instrument, and on July 5, 2004, on the instrument used to perform the mercury analyses. The analytical methods specify that IDLs must be established within three months of the associated sample analyses.

Linear ranges were established on October 4, 2004, on the instrument used to perform ICP-MS analyses and on January 27, 2005, on the instrument used to perform the ICP-AES analyses. Method 6020 does not specify a required frequency for the establishment of linear ranges, but Method 6010B specifies that linear ranges must be established within three months of the associated sample analyses.

Interelement correction factors were performed on December 31, 2003, on the instrument used to perform ICP-AES analyses. Method 6010B specifies that interelement correction factors must be established within one year of the associated sample analyses. Interelement correction factors are not required for analyses performed by ICP-MS.

Based on professional judgment, as concentrations approach the IDL the accuracy of the measurement decreases; values closer to the EQL, however, are probably quite accurate. Therefore, a guideline of two times the IDL was used to determine whether the reported results warranted qualification. Sample results below the respective reporting limit, less than two times the IDL, and not otherwise qualified were qualified as estimated (J). Specifically, results for silver in A2-3-S1, A2-3-S1D, and A1-3-S1-2, for selenium in A2-3-S1D, A2-13-S1, A1-26-S1, A1-3-S1, and A1-3-S1-2, and for thallium in A2-13-S1 and A1-3-S1-2 were so qualified.

## **XI. Documentation**

Chain of custody records were present and accurately completed for all samples reported in this data package with the following exceptions:

- The samples are not listed on the chain of custody record in chronological order suggesting that this document was not completed as the samples were collected.
- Improper editing was noted on the chain of custody record. When necessary, corrections must be made by drawing a single line through the error, entering the correct information, and initialing and dating the correction.
- The chain of custody record and laboratory cooler receipt log indicate that no custody seals were used on the sample cooler.

With regard to data presentation:

- The chain of custody record for the sample transfer to the Kimberly, Wisconsin laboratory was not included in the data package. The laboratory provided the missing document on April 4, 2005, and it should be included in the original and all copies of the data package to ensure that accurate and complete documentation is available for future reference.

These chain of custody and data presentation issues do not directly affect the validity of the reported results, but they could be problematic if these data are used in litigation.

## **XII. Overall Assessment**

Findings of the validation effort resulted in the following qualifications of sample results:

- Based on unacceptable recoveries in one or both matrix spike analyses, results for antimony, cadmium, and nickel in all of the samples in this data set were qualified as estimated (UJ, J). Results for nickel also warranted estimation based on an unacceptable serial dilution result.
- Based on poor precision in the triplicate analyses, results for zinc in all of the samples in this data set were qualified as estimated (J).
- Based on unacceptable results in the serial dilution analyses, results for aluminum, copper, chromium, magnesium, and potassium in all of the soil samples in this data set were qualified as estimated (J).
- Based on professional judgment, results for silver in A2-3-S1, A2-3-S1D, and A1-3-S1-2, for selenium in A2-3-S1D, A2-13-S1, A1-26-S1, A1-3-S1, and A1-3-S1-2, and for thallium in A2-13-S1 and A1-3-S1-2 were qualified as estimated (J) because the reported concentrations were less than two times the instrument detection limit and not otherwise qualified.

The validator removed all “A,” “B,” “C,” “E,” “N,” and “\*” qualifiers applied by the laboratory.

Chain of custody documentation and data presentation issues are discussed in Section XI.

This validation report should be considered part of the data package for all future distributions of the inorganics data.



**ATTACHMENT A**

**DATA SUMMARY FORM**

**Target Analyte List Metals in Soil Samples  
Sample Delivery Group Number 857185**

DATA SUMMARY FORM: INORGANICS  
SOIL SAMPLES  
(mg/kg)

Site Name: Eagle Zinc

Sampling Date: March 16, 2005

Laboratory Sample Delivery Group Number: 857185

Trillium Project No.: 05906

Sample Number		A2-3-S1	A2-3-S1D	A2-13-S1	A1-26-S1	A1-3-S1	A1-3-S1-2		
Lab ID		857185-001	857185-002	857185-003	857185-004	857185-005	857185-005		
Percent Solids		80.8	81.0	84.0	82.0	84.4	83.4		
EQL	Analyte								
15	Aluminum	11000 J	11000 J	9800 J	19000 J	18000 J	21000 J		
7.6	Antimony	19 UJ	18 UJ	18 UJ	18 UJ	5.4 J	1.8 UJ		
0.30	Arsenic	11	7.4	2.3	12	21	4.5		
0.30	Barium	160	150	150	190	150	110		
0.10	Beryllium	0.78	0.65	0.65	0.80	0.71	1.0		
0.10	Cadmium	7.7 J	7.3 J	5.8 J	7.3 J	7.8 J	4.7 J		
30	Calcium	650	670	1800	1000	1000	1600		
0.30	Chromium	15 J	15 J	13 J	21 J	22 J	23 J		
0.20	Cobalt	18	8.0	3.3	13	12	6.0		
1.0	Copper	7.7 J	12 J	27 J	130 J	180 J	12 J		
30	Iron	16000	12000	8100	27000	25000	19000		
0.25	Lead	30	29	26	500	1100	24		
15	Magnesium	1400 J	1400 J	990 J	2200 J	2700 J	2500 J		
0.20	Manganese	960	400	160	540	490	190		
0.100	Mercury	0.020	0.023	0.034	0.042	0.028	0.041		
0.30	Nickel	11 J	9.2 J	8.0 J	42 J	18 J	16 J		
20	Potassium	900 J	940 J	840 J	1300 J	1400 J	670 J		
1.0	Selenium	1.2	0.6 J	0.81 J	0.99 J	11 J	0.64 J		
0.30	Silver	0.06 J	0.05 J	0.10	0.97	3.4	0.05 J		
50	Sodium	70	66	98	53	41	73		
0.30	Thallium	0.35	0.27	0.19 J	0.35	0.31	0.17 J		
0.30	Vanadium	40	33	23	39	42	33		
10	Zinc	460 J	710 J	770 J	4800 J	2700 J	93 J		



**ATTACHMENT B**

**LABORATORY ANSWER FORMS**

**Target Analyte List Metals in Soil Samples  
Sample Delivery Group Number 857185**

# En Chem

A Division of Pace Analytical Services, Inc.

## Analytical Report Number: 857185

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number :

Field ID : A2-3-S1

Matrix Type : SOIL

Collection Date : 03/16/05

Report Date : 03/31/05

Lab Sample Number : 857185-001

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	11000 J	19	5	mg/Kg	<del>E</del>	03/19/05	SW846 3050B	SW846 6020
Antimony	< 19 JJ	19	10	mg/Kg	<del>D</del>	03/22/05	SW846 3050B	SW846 6010B
Arsenic	11	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Barium	160	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Beryllium	0.78	0.12	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cadmium	7.7 J	0.12	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Calcium	650	37	5	mg/Kg	<del>EA</del>	03/19/05	SW846 3050B	SW846 6020
Chromium	15 J	0.37	5	mg/Kg	<del>E</del>	03/19/05	SW846 3050B	SW846 6020
Cobalt	18	0.25	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Copper	7.7 J	1.2	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Iron	16000	37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Lead	30	0.31	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Magnesium	1400 J	19	5	mg/Kg	<del>E</del>	03/19/05	SW846 3050B	SW846 6020
Manganese	960	0.25	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Mercury	0.020	0.012	1	mg/kg	<del>A</del>	03/23/05	SW846 7471A	SW846 7471A
Nickel	11 J	0.37	5	mg/Kg	<del>E</del>	03/19/05	SW846 3050B	SW846 6020
Potassium	900 J	25	5	mg/Kg	<del>E</del>	03/19/05	SW846 3050B	SW846 6020
Selenium	1.2	1.2	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Silver	0.056 J X	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Sodium	70	62	5	mg/Kg	<del>A</del>	03/19/05	SW846 3050B	SW846 6020
Thallium	0.35 J	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Vanadium	40	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Zinc	460 J	25	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	80.8	---	1	%		03/24/05	SM 2540G M	SM 2540G M

see 04/05/05

**En Chem**

A Division of Pace Analytical Services, Inc.

**Analytical Report Number: 857185**1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number :

Field ID : A2-3-S1D

Matrix Type : SOIL

Collection Date : 03/16/05

Report Date : 03/31/05

Lab Sample Number : 857185-002

**INORGANICS**

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	11000 <b>J</b>	19	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Antimony	< 18 <b>43</b>	18	10	mg/Kg	<del>✓</del>	03/22/05	SW846 3050B	SW846 6010B
Arsenic	7.4	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Barium	150	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Beryllium	0.65	0.12	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cadmium	7.3 <b>J</b>	0.12	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Calcium	670	37	5	mg/Kg	<del>✓</del>	03/19/05	SW846 3050B	SW846 6020
Chromium	15 <b>J</b>	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cobalt	8.0	0.25	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Copper	12 <b>J</b>	1.2	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Iron	12000	37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Lead	29	0.31	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Magnesium	1400 <b>J</b>	19	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Manganese	400	0.25	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Mercury	0.023	0.012	1	mg/kg	<del>✓</del>	03/23/05	SW846 7471A	SW846 7471A
Nickel	9.2 <b>J</b>	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Potassium	940 <b>J</b>	25	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Selenium	0.88 <b>J</b> <del>✓</del>	1.2	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Silver	0.050 <b>J</b> <del>✓</del>	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Sodium	66	62	5	mg/Kg	<del>✓</del>	03/19/05	SW846 3050B	SW846 6020
Thallium	0.27 <del>✓</del>	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Vanadium	33	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Zinc	710 <b>J</b>	25	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	81.0	—	1	%		03/24/05	SM 2540G M	SM 2540G M

en 04/05/05

# En Chem

## Analytical Report Number: 857185

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number :

Field ID : A2-13-S1

Matrix Type : SOIL

Collection Date : 03/16/05

Report Date : 03/31/05

Lab Sample Number : 857185-003

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	9800 J	18	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Antimony	< 18 uJ	18	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Arsenic	2.3	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Barium	150	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Beryllium	0.65	0.12	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cadmium	5.8 J	0.12	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Calcium	1800	36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Chromium	13 J	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cobalt	3.3	0.24	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Copper	27 J	1.2	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Iron	8100	36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Lead	26	0.30	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Magnesium	990 J	18	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Manganese	160	0.24	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Mercury	0.034	0.012	1	mg/kg	A	03/23/05	SW846 7471A	SW846 7471A
Nickel	8.0 J	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Potassium	840 J	24	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Selenium	0.81 J	1.2	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Silver	0.10	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Sodium	98	59	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Thallium	0.19 J	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Vanadium	23	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Zinc	770 J	24	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	84.0	---	1	%		03/24/05	SM 2540G M	SM 2540G M

in 04/05/05



**En Chem****Analytical Report Number: 857185**1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number :

Field ID : A1-26-S1

Matrix Type : SOIL

Collection Date : 03/16/05

Report Date : 03/31/05

Lab Sample Number : 857185-004

**INORGANICS**

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	19000 J	180	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Antimony	< 18 uJ	18	10	mg/Kg	CK	03/22/05	SW846 3050B	SW846 6010B
Arsenic	12	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Barium	190	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Beryllium	0.80	0.12	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cadmium	7.3 J	0.12	5	mg/Kg	X	03/19/05	SW846 3050B	SW846 6020
Calcium	1000	37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Chromium	21 J	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cobalt	13	0.24	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Copper	130 J	1.2	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Iron	27000	370	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Lead	500	3.1	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Magnesium	2200 J	18	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Manganese	540	0.24	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Mercury	0.042	0.012	1	mg/kg	X	03/23/05	SW846 7471A	SW846 7471A
Nickel	42 J	0.37	5	mg/Kg	X	03/19/05	SW846 3050B	SW846 6020
Potassium	1300 J	24	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Selenium	0.99 J	1.2	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Silver	0.97	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Sodium	53	61	5	mg/Kg	X	03/19/05	SW846 3050B	SW846 6020
Thallium	0.35	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Vanadium	39	0.37	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Zinc	4800 J	240	100	mg/Kg	/	03/22/05	SW846 3050B	SW846 6010B
Percent Solids	82.0	—	1	%		03/24/05	SM 2540G M	SM 2540G M

u 04/05/05

# En Chem

## Analytical Report Number: 857185

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number :

Field ID : A1-3-S1

Matrix Type : SOIL

Collection Date : 03/16/05

Report Date : 03/31/05

Lab Sample Number : 857185-005

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	18000 J	180	50	mg/Kg		03/21/05	SW846 3050B	SW846 6020
Antimony	5.4 J	18	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Arsenic	21	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Barium	150	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Beryllium	0.71	0.12	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cadmium	7.8 J	0.12	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Calcium	1000	36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Chromium	22 J	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cobalt	12	0.24	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Copper	180 J	1.2	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Iron	25000	36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Lead	1100	0.30	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Magnesium	2700 J	18	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Manganese	490	0.24	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Mercury	0.028	0.012	1	mg/kg		03/23/05	SW846 7471A	SW846 7471A
Nickel	18 J	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Potassium	1400 J	24	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Selenium	1.1 J	1.2	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Silver	3.4	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Sodium	41	59	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Thallium	0.31	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Vanadium	42	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Zinc	2700 J	24	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	84.4	---	1	%		03/24/05	SM 2540G M	SM 2540G M

en 04/05/05

# En Chem

## Analytical Report Number: 857185

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number :

Field ID : A1-3-S1-2

Matrix Type : SOIL

Collection Date : 03/16/05

Report Date : 03/31/05

Lab Sample Number : 857185-006

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	21000 J	180	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Antimony	< 1.8 JJ	1.8	1	mg/Kg	C	03/22/05	SW846 3050B	SW846 6010B
Arsenic	4.5	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Barium	110	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Beryllium	1.0	0.12	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cadmium	4.7 J	0.12	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Calcium	1600	36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Chromium	23 J	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cobalt	6.0	0.24	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Copper	12 J	1.2	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Iron	19000	36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Lead	24	0.30	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Magnesium	2500 J	18	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Manganese	190	0.24	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Mercury	0.041	0.012	1	mg/kg	A	03/23/05	SW846 7471A	SW846 7471A
Nickel	16 J	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Potassium	670 J	24	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Selenium	0.64 J X	1.2	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Silver	0.054 J X	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Sodium	73	60	5	mg/Kg	A	03/19/05	SW846 3050B	SW846 6020
Thallium	0.17 J X	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Vanadium	33	0.36	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Zinc	93 J	2.4	1	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	83.4	---	1	%		03/24/05	SM 2540G M	SM 2540G M

see 04/05/05



**DATA VALIDATION**  
**FOR**  
**EAGLE ZINC PROJECT**

**INORGANICS ANALYSIS DATA**  
**Target Analyte List Metals in Soil Samples**  
**Samples Collected March 11, 2005**

**Chemical Analyses Performed by:**  
**EnChem (Division of Pace Analytical Services, Inc.)**  
**Green Bay, Wisconsin (All Analytes except Mercury)**  
**Kimberly, Wisconsin (Mercury)**  
**Sample Delivery Group Number 857072B**

**Data Deliverables Prepared for:**  
**ENVIRON International Corporation**  
**Deerfield, Illinois**

**Data Validation Performed by:**  
**Trillium, Inc.**  
**9312 Highland Gardens Road**  
**Baton Rouge, Louisiana 70811**  
**(225) 355-8702**  
**(225) 355-8987 (Fax)**

**April 5, 2005**

## EXECUTIVE SUMMARY

Validation of the inorganics analysis data [target analyte list (TAL) metals] was completed by Trillium, Inc. Analyses for mercury were performed by EnChem Laboratories in Kimberly, Wisconsin. Analyses for the remaining target analytes were performed by EnChem Laboratories in Green Bay, Wisconsin. The samples in this data set represent the sample collections from March 11, 2005, from the Eagle Zinc Project. Data for nine soil samples were reported by the two laboratories in a single data package under Sample Delivery Group (SDG) 857072B, which was received for review on April 1, 2005. The data package includes the following samples:

---

RR1-2	RR1-1	MP1-21	NA-S1	NA-S2	NA-S3
NA-S4	NA-S2	RRO-12D			

---

Findings of the validation effort resulted in the following qualifications of sample results:

- Results for antimony, chromium, magnesium, manganese, and potassium in all of the samples in this data set were qualified as estimated (UJ, J).
- Results for thallium in RR1-2, MP1-21, NA-S2D, NA-S4, and RRO-12D, for selenium in NA-S1, NA-S2, NA-S3, NA-S4, and NA-S2D, and for silver in NA-S4 were qualified as estimated (J).

Brief explanations of the reasons for the actions taken above may be found in the Overall Assessment (Section XII). Details of the validation findings and conclusions based on the review of the results for each quality control requirement are provided in the remaining sections of this report.

The validator removed all "A," "B," "C," "E," "N," and "\*" qualifiers applied by the laboratory.

Chain of custody documentation issues are discussed in Section XI.

This validation report should be considered part of the data package for all future distributions of the inorganics data.

## INTRODUCTION

Analyses were performed according to EPA Methods 3050B, 7471A, 6010B, and 6020 (SW-846; Test Methods for Evaluating Solid Waste, Third Edition, through Update III, 12/96). Results of sample analyses are reported by the laboratory as either qualified or unqualified; various qualifier codes are used by the laboratory to denote specific information regarding the analytical results. In this data set, the laboratory used the following defined qualifiers:

- A Analyte is detected in the method blank. Method blank criteria are evaluated to the laboratory method detection limit. Additionally, method blank acceptance may be based on project specific criteria or determined from analyte concentrations in the sample and are evaluated on a sample by sample basis.
- B The analyte has been detected between the method detection limit and the reporting limit.
- C Elevated detection limit.
- E Estimated concentration due to matrix interferences. During the metals analysis the serial dilution failed to meet the established control limits of 0-10%. The sample concentration is greater than 50 times the IDL for analysis done on the ICP or 100 times the IDL for analysis done on the ICP-MS. The result was flagged with the E qualifier to indicate that a physical interference was observed.
- N Spiked sample recovery not within control limits.
- \* Precision not within control limits.

The validator removed all "A," "B," "C," "E," "N," and "\*" qualifiers applied by the laboratory.

To the extent applicable, Trillium's validation was performed in conformance with the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (EPA 540-R-01-008, July 2002). Where discrepancies were found, the specifications of the referenced methods took precedence. In addition, professional judgment was applied as necessary and appropriate.

The data validation process is intended to evaluate data on a technical basis rather than a contract compliance basis. This requires that the data package contain sufficient raw data documentation to facilitate the validation process and allow verification of all reported results. It is also assumed that the data package represents the best efforts of the laboratory and has already been subjected to adequate and sufficient quality review prior to submission for validation.

During the validation process, laboratory data are verified against all available supporting documentation. Based on this evaluation, qualifier codes may be added, deleted, or modified by the data validator. Validated results are, therefore, either qualified or unqualified. Unqualified results mean that the reported values may be used without reservation. Final validated results are annotated with the following codes as defined in the National Functional Guidelines:

- U      The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
- J      The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
- J+     The result is an estimated quantity, but the result may be biased high.
- J-     The result is an estimated quantity, but the result may be biased low.
- R      The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.
- UJ     The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

These codes are recorded on the Data Summary Forms contained in Attachment A, as well as on the laboratory answer forms in Attachment B of this validation report to qualify the results as appropriate according to the review of the data package.

Two facts should be noted by all data users. First, **the “R” qualifier means that the laboratory-reported value is unusable.** In other words, due to significant quality control problems, the analysis is invalid and provides no information as to whether the compound is present or not. Rejected values should not appear on data tables because they cannot be relied upon, even as a last resort. Second, **no analyte concentration is guaranteed to be accurate even if all associated quality control is acceptable.** Strict quality control conformance serves only to increase confidence in reported results; any analytical result will always contain some error.

The data user is also cautioned that the validation effort is based on the raw data printouts as provided by the laboratory. Software manipulation cannot be routinely detected during validation; unless otherwise stated in the report, these kinds of issues are outside the scope of this review.

## **I. Holding Times, Preservation and Sample Integrity**

The soil samples were collected on March 11, 2005. All of the samples were digested and analyzed well within acceptable holding times (28 days for mercury and 180 days for the remaining analytes).

The temperature of the sample cooler (1°C), as recorded on the Chain of Custody Record and the Cooler Receipt Log, was slightly below the acceptable temperature range (4°C  $\pm$  2°C). Based on professional judgment, no adverse effect on the metals results would be expected based on the slightly low cooler temperature and no data were qualified on this basis.

The Chain of Custody Record and Cooler Receipt Log also indicated that the samples were received intact but no custody seals were used on the sample cooler.

Copies of the FedEx airbills were provided by the laboratory on April 4, 2005, to document custody of the samples between the field and the laboratory.

## **II. Mass Spectrometer Tune**

The tuning solution for the ICP-MS was analyzed on March 19, 2005 at 16:18. All criteria specified in Method 6020 were met.

As required by the analytical method, a blank and three calibration standards were analyzed to document instrument stability. The laboratory confirmed that three integrations of each target analyte were performed but only the average of these integrations was reported in the supporting data. Percent relative standard deviation (%RSD) values were reported by the laboratory but could not be confirmed by the validator since abbreviated raw data were provided. No acceptance limits are provided in Method 6020 but all reported %RSD values were less than 10%, which was deemed acceptable based on professional judgment.

## **III. Calibrations**

Inductively coupled plasma-mass spectrometry (ICP-MS) analyses were performed on an instrument identified as "HP ICPMS 2." The samples reported in this data set were analyzed in two analysis series on March 19 and 21, 2005.

Inductively coupled plasma-atomic emission spectroscopy (ICP-AES) analyses were performed on a trace ICP-AES instrument identified as "TJA61E Trace ICP 3." The samples reported in this data set were analyzed in a single analysis series on March 22, 2005.

Mercury analyses were performed in a single analysis series run on March 17, 2005 on a cold vapor atomic absorption (CVAA) system identified as "PE CVAA 1."





Initial calibration verification (ICV) standards were performed for all of the target analytes after each initial calibration. All ICV percent recoveries (%R) were correctly calculated, accurately reported, and within the validation guideline-specified control limits (90-110% for analytes determined by ICP-MS and ICP-AES and 80-120% for mercury).

Continuing calibration verification (CCV) standards were run at the appropriate frequency (after every ten samples) in all of the analysis series. All %Rs were correctly calculated, accurately reported, and within the method- and validation guideline-specified control limits (90-110% for analytes determined by ICP-MS and ICP-AES and 80-120% for mercury).

A linearity check for mercury was performed at the start of the analysis series and gave an acceptable correlation coefficient ( $>0.995$ ).

The analysis of "CRI" standards in the ICP-MS and ICP-AES analyses and "CRA" standards in the mercury analyses are not required by SW-846 methods and were not performed in association with the reported analyses.

#### **IV. Blanks**

Initial and continuing calibration blanks (ICBs and CCBs) were analyzed at the appropriate frequencies throughout each analytical sequence. No target analytes were detected in any of the ICBs or CCBs.

Aluminum (11.925 mg/kg), calcium (28.545 mg/kg), chromium (0.175 mg/kg), copper (0.080 mg/kg), iron (29.319 mg/kg), lead (0.195 mg/kg), magnesium (8.855 mg/kg), manganese (0.130 mg/kg), potassium (10.010 mg/kg), sodium (21.845 mg/kg), and zinc (0.832 mg/kg) were detected in the solid-matrix preparation blank (PB). With regard to PBs, the validation guidance document indicates that if an analyte is detected in the PB below the contract required quantitation limit (CRQL), "no correction of the sample results should be performed." For these samples, the estimated quantitation limit (EQL) was substituted for the CRQL. None of the analytes in the PB were detected at concentrations greater than the analyte-specific EQL; therefore, no data were qualified on this basis. In addition, results for these analytes in the site samples were significantly greater than the concentrations detected in the PB (i.e., with the exceptions of sodium in MP1-21, NA-S1, NA-S2, NA-S3, NA-S4, and NA-S2D, all were greater than five times the concentration detected in the PB). The data user is cautioned in the use of the results reported for sodium in MP1-21, NA-S1, NA-S2, NA-S3, NA-S4, and NA-S2D, as it is possible that this analyte was artificially introduced in these samples and may not be a true sample component.

No field-submitted blanks were included in this data set.

## **V. ICP Interference Check Sample**

ICP interference check sample analyses were performed at the appropriate frequency throughout all analysis series. Target analyte recoveries were acceptable (80-120%) in all cases.

## **VI Spiked Sample Analyses**

### **A. Laboratory Control Samples**

A solid-matrix laboratory control sample pair (LCS/LCSD) was prepared and analyzed with each analysis series. Each LCS/LCSD pair included all of the target analytes required for that analytical sequence. All percent recovery (%R) and relative percent difference (RPD) values were correctly calculated, accurately reported, and within the analyte-specific acceptance limits specified by the laboratory on the summary forms.

### **B. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analyses**

NA-S3 was prepared as an MS/MSD pair for all of the target analytes. All %R and RPD values were correctly calculated and accurately reported. For the target analytes with an amount of spike added greater than four times the concentration of the analyte in the original analysis of NA-S3, %Rs and RPDs were within the analyte-specific acceptance limits specified by the laboratory on the summary forms with the following exception:

Analyte	MS	MSD	Acceptance Limits
antimony	64%	59%	80-120%

Based on unacceptably low recoveries in both of the matrix spike analyses, results for antimony in all of the samples in this data set qualified as estimated (UJ, J).

For the target analytes with an amount of spike added less than four times the concentration of the analyte detected in the original analysis of NA-S3, acceptable recoveries would not be expected. Since the amount of spike added is insignificant, this presents an opportunity to compare precision among the triplicate analyses (sample, MS, and MSD). Based on professional judgment, an acceptance limit of  $\leq 25\%$  RSD was used to evaluate the precision among results reported for aluminum, barium, iron, manganese, and zinc. With the exception of manganese (31.8%), acceptable precision among the triplicate results was observed (1.9% to 3.8%). Based on poor precision in the triplicate analyses, results for manganese in all of the samples in this data set were qualified as estimated (J).

Post digestion spike recoveries for all of the TAL target analytes in NA-S3 were acceptable (75-125% acceptance limits).

## **VII. Duplicate Sample Analyses**

### **A. Laboratory Duplicate Analysis**

No laboratory duplicate analysis was performed on a sample in this data set.

### **B. Field Duplicate Analyses**

No field duplicate pair was identified in this sample set.

## **VIII Internal Standard Performance**

Lithium (mass 6), scandium (mass 45), germanium (mass 74), yttrium (mass 89), indium (mass 115), terbium (mass 159), and bismuth (mass 209) were included as internal standard analytes for the ICP-MS analyses. Percent recoveries of the internal standards were within the analytical method-specified 30-120% acceptance limits in all of the site and quality control sample analyses.

## **IX ICP Serial Dilution**

ICP-AES serial dilution analysis for antimony and zinc was performed on RR0-12D. Results for elements with initial (undiluted) results greater than 10 times the instrument detection limit were acceptable (<10% Difference).

ICP-MS serial dilution analysis was performed on RR1-2. Results for elements with initial (undiluted) results greater than 100 times the concentration of the analyte in the reagent blank were acceptable (<10% Difference) with the exceptions of chromium (15%), magnesium (16%), and potassium (12%). Based on unacceptable results in the serial dilution analysis, results for chromium, magnesium, and potassium in all of the soil samples in this data set were qualified as estimated (J).

## **X. Sample Results Verification**

For the ICP-MS analyses, the laboratory confirmed that three integrations of each target analyte were performed but only the average of these integrations was reported in the supporting data; therefore, the reported average concentration could not be confirmed by the validator. Based on the reported average concentrations for the analytes determined by ICP-MS and for all remaining target analytes, all sample results were correctly calculated and accurately reported from the raw data, including adjustments for the solid-matrix preparation procedures, dilutions, and percent solids.



Instrument detection limits (IDLs) were reported for all three instruments used to perform the reported sample results. IDLs were established on April 4, 2004, on the ICP-MS instrument, on January 5, 2004, on the ICP-AES instrument, and on July 5, 2004, on the instrument used to perform the mercury analyses. The analytical methods specify that IDLs must be established within three months of the associated sample analyses.

Linear ranges were established on October 4, 2004, on the instrument used to perform ICP-MS analyses and on January 27, 2005, on the instrument used to perform the ICP-AES analyses. Method 6020 does not specify a required frequency for the establishment of linear ranges, but Method 6010B specifies that linear ranges must be established within three months of the associated sample analyses.

Interelement correction factors were performed on December 31, 2003, on the instrument used to perform ICP-AES analyses. Method 6010B specifies that interelement correction factors must be established within one year of the associated sample analyses. Interelement correction factors are not required for analyses performed by ICP-MS.

Based on professional judgment, as concentrations approach the IDL the accuracy of the measurement decreases; values closer to the EQL, however, are probably quite accurate. Therefore, a guideline of two times the IDL was used to determine whether the reported results warranted qualification. Sample results below the respective reporting limit, less than two times the IDL, and not otherwise qualified were qualified as estimated (J). Specifically, results for thallium in RR1-2, MP1-21, NA-S2D, NA-S4, and RRO-12D, for selenium in NA-S1, NA-S2, NA-S3, NA-S4, and NA-S2D, and for silver in NA-S4 were so qualified.

## **XI. Documentation**

Chain of custody records were present and accurately completed for all samples reported in this data package with the following exceptions:

- The samples are not listed on the chain of custody record in chronological order suggesting that this document was not completed as the samples were collected.
- The chain of custody record indicates these samples were “relinquished by” Ross Jones on March 12, 2005, but does not indicate a “received by” signature, date, or time. A “relinquished by” FedEx is indicated on March 14, 2005, and a laboratory “received by” signature indicates the samples were received on March 14, 2005, at 09:20. A copy of the FedEx Airbill was requested, which indicated the samples were shipped on March 12, 2005, were in transit with FedEx on March 13, 2005, and were delivered on March 14, 2005. A copy of the airbill should be included in the original and all copies of the data package to ensure that complete documentation is available for future reference.



- The chain of custody record and laboratory cooler receipt log indicate that no custody seals were used on the sample cooler.

No data presentation problems were noted in the data package received for review.

These chain of custody issues do not directly affect the validity of the reported results, but they could be problematic if these data are used in litigation.

## **XII. Overall Assessment**

Findings of the validation effort resulted in the following qualifications of sample results:

- Based on unacceptably low recoveries in both matrix spike analyses, results for antimony in all of the samples in this data set were qualified as estimated (UJ, J).
- Based on poor precision in the triplicate analyses, results for manganese in all of the samples in this data set were qualified as estimated (J).
- Based on unacceptable results in the serial dilution analysis, results for chromium, magnesium, and potassium in all of the soil samples in this data set were qualified as estimated (J).
- Based on professional judgment, results for thallium in RR1-2, MP1-21, NA-S2D, NA-S4, and RRO-12D, for selenium in NA-S1, NA-S2, NA-S3, NA-S4, and NA-S2D, and for silver in NA-S4 were qualified as estimated (J) because the reported concentrations were less than two times the instrument detection limit and not otherwise qualified.

Chain of custody documentation issues are discussed in Section XI.

This validation report should be considered part of the data package for all future distributions of the inorganics data.



**ATTACHMENT A**

**DATA SUMMARY FORMS**

**Target Analyte List Metals in Soil Samples  
Sample Delivery Group Number 857072B**

DATA SUMMARY FORM: INORGANICS  
SOIL SAMPLES  
(mg/kg)

Site Name: Eagle Zinc

Sampling Date: March 11, 2005

Laboratory Sample Delivery Group Number: 857072B

Trillium Project No.: 05906

Sample Number Lab ID		RR1-2	NP-15	MP1-21	NA-S1	NA-S2	NA-S3	NA-S4	NA-S2D
Percent Solids		857072-013	857072-014	857072-015	857072-016	857072-017	857072-018	857072-019	857072-020
EQL Analyte		94.1	95.2	93.6	77.5	79.9	75.8	74.5	72.4
15	Aluminum	7300	5300	5700	11000	8400	11000	7600	8600
7.6	Antimony	16 UJ	16 UJ	190 J	19 UJ	19 UJ	20 UJ	20 UJ	21 UJ
0.30	Arsenic	6.8	9.1	200	7.3	4.0	3.7	3.0	4.8
0.30	Barium	130	160	870	160	120	150	84	93
0.10	Beryllium	0.79	1.1	0.84	0.56	0.46	0.53	0.38	0.58
0.10	Cadmium	9.4	5.6	50	2.5	5.9	2.7	1.5	7.7
30	Calcium	3500	6200	2100	8500	1100	2300	1700	1500
0.30	Chromium	9.2 J	8.6 J	22 J	14 J	11 J	13 J	9.7 J	13 J
0.20	Cobalt	70	140	110	8.3	4.2	3.7	2.9	6.6
1.0	Copper	2000	3400	3600	20	67	19	10	170
30	Iron	60000	75000	110000	14000	9000	11000	7300	10000
0.25	Lead	250	450	31000	87	120	40	31	230
15	Magnesium	1400 J	3400 J	1000 J	1300 J	1000 J	1200 J	920 J	1100 J
0.20	Manganese	190 J	330 J	8300 J	1000 J	260 J	260 J	280 J	320 J
0.100	Mercury	0.038	0.053	0.065	0.020	0.031	0.019	0.015	0.050
0.30	Nickel	610	790	59	11	11	9.6	6.6	37
20	Potassium	490 J	770 J	140 J	910 J	730 J	870 J	810 J	750 J
1.0	Selenium	4.7	5.7	4.7	0.89 J	0.88 J	0.59 J	0.62 J	1.1 J
0.30	Silver	3.9	8.9	140	0.26	0.22	0.11	0.10 J	0.38
50	Sodium	200	230	51	36	47	37	33	58
0.30	Thallium	0.05 J	0.32 UJ	0.11 J	0.20	0.17	0.16	0.13 J	0.17 J
0.30	Vanadium	12	12	21	32	21	28	19	22
10	Zinc	190000	210000	39000	1600	5100	1500	950	7700

DATA SUMMARY FORM: INORGANICS  
SOIL SAMPLES  
(mg/kg)

Site Name: Eagle Zinc

Sampling Date: March 11, 2005

Laboratory Sample Delivery Group Number: 857072B

Trillium Project No.: 05906

Sample Number Lab ID Percent Solids		RRO-12D															
		857072-021															
		90.0															
EQL	Analyte																
15	Aluminum	11000															
7.6	Antimony	17	UJ														
0.30	Arsenic	15															
0.30	Barium	420															
0.10	Beryllium	2.0															
0.10	Cadmium	10															
30	Calcium	19000															
0.30	Chromium	38	J														
0.20	Cobalt	560															
1.0	Copper	3400															
30	Iron	73000															
0.25	Lead	520															
15	Magnesium	5200	J														
0.20	Manganese	1300	J														
0.100	Mercury	0.047															
0.30	Nickel	1100															
20	Potassium	1300	J														
1.0	Selenium	5.5															
0.30	Silver	34															
50	Sodium	1700															
0.30	Thallium	0.05	J														
0.30	Vanadium	20															
10	Zinc	150000															





**ATTACHMENT B**

**LABORATORY ANSWER FORMS**

**Target Analyte List Metals in Soil Samples  
Sample Delivery Group Number 857072B**

# En Chem

## Analytical Report Number: 857072B

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : RR1-2

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/31/05

Lab Sample Number : 857072-013

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	7300	16	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Antimony	< 16 <b>uj</b>	16	10	mg/Kg	<del>E</del>	03/22/05	SW846 3050B	SW846 6010B
Arsenic	6.8	0.32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Barium	130	0.32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Beryllium	0.79	0.11	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cadmium	9.4	0.11	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Calcium	3500	32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Chromium	9.2 <b>J</b>	0.32	5	mg/Kg	<del>E</del>	03/19/05	SW846 3050B	SW846 6020
Cobalt	70	0.21	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Copper	2000	1.1	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Iron	60000	320	50	mg/Kg		03/21/05	SW846 3050B	SW846 6020
Lead	250	0.27	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Magnesium	1400 <b>J</b>	16	5	mg/Kg	<del>E</del>	03/19/05	SW846 3050B	SW846 6020
Manganese	190 <b>J</b>	0.21	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Mercury	0.038	0.011	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	610	0.32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Potassium	490 <b>J</b>	21	5	mg/Kg	<del>E</del>	03/19/05	SW846 3050B	SW846 6020
Selenium	4.7	1.1	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Silver	3.9	0.32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Sodium	200	53	5	mg/Kg	<del>A</del>	03/19/05	SW846 3050B	SW846 6020
Thallium	0.053 <b>J</b>	0.32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Vanadium	12	0.32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Zinc	190000	210	100	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	94.1	---	1	%		03/15/05	SM 2540G M	SM 2540G M

*ew 04/05/05*

**En Chem**

A Division of Pace Analytical Services, Inc.

**Analytical Report Number: 857072B**1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : RR1-1

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/31/05

Lab Sample Number : 857072-014

**INORGANICS**

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	5300	16	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Antimony	< 16 <b>UJ</b>	16	10	mg/Kg	<del>Q</del>	03/22/05	SW846 3050B	SW846 6010B
Arsenic	9.1	0.32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Barium	160	0.32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Beryllium	1.1	0.11	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cadmium	5.6	0.11	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Calcium	6200	32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Chromium	8.6 <b>J</b>	0.32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cobalt	140	0.21	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Copper	3400	1.1	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Iron	75000	320	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Lead	450	0.26	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Magnesium	3400 <b>J</b>	16	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Manganese	330 <b>J</b>	0.21	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Mercury	0.053	0.011	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	790	0.32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Potassium	770 <b>J</b>	21	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Selenium	5.7	1.1	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Silver	8.9	0.32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Sodium	230	53	5	mg/Kg	<del>A</del>	03/19/05	SW846 3050B	SW846 6020
Thallium	< 0.32	0.32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Vanadium	12	0.32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Zinc	210000	210	100	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	95.2	---	1	%		03/15/05	SM 2540G M	SM 2540G M

*see 04/05/05*

# En Chem

Analytical Report Number: 857072B

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : MP1-21

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/31/05

Lab Sample Number : 857072-015

## INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	5700	160	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Antimony	190 J	16	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Arsenic	200	3.2	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Barium	870	3.2	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Beryllium	0.84	0.11	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cadmium	50	1.1	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Calcium	2100	32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Chromium	22 J	3.2	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cobalt	110	2.1	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Copper	3600	11	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Iron	110000	320	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Lead	31000	2.7	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Magnesium	1000 J	16	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Manganese	8300 J	2.1	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Mercury	0.065	0.011	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	59	3.2	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Potassium	140 J	21	5	mg/Kg	X	03/19/05	SW846 3050B	SW846 6020
Selenium	4.7	11	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Silver	140	3.2	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Sodium	51	53	5	mg/Kg	X	03/19/05	SW846 3050B	SW846 6020
Thallium	0.11 J	0.32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Vanadium	21	3.2	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Zinc	39000	21	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	93.6	---	1	%		03/15/05	SM 2540G M	SM 2540G M

me 04/05/05

# En Chem

## Analytical Report Number: 857072B

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : NA-S1

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/31/05

Lab Sample Number : 857072-016

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	11000	19	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Antimony	< 19 <b>JS</b>	19	10	mg/Kg	<del>JS</del>	03/22/05	SW846 3050B	SW846 6010B
Arsenic	7.3	0.39	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Barium	160	0.39	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Beryllium	0.56	0.13	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cadmium	2.5	0.13	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Calcium	8500	39	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Chromium	14 <b>J</b>	0.39	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cobalt	8.3	0.26	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Copper	20	1.3	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Iron	14000	39	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Lead	87	0.32	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Magnesium	1300 <b>J</b>	19	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Manganese	1000 <b>J</b>	0.26	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Mercury	0.020	0.013	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	11	0.39	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Potassium	910 <b>J</b>	26	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Selenium	0.89 <b>J</b> <del>JS</del>	1.3	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Silver	0.26 <del>JS</del>	0.39	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Sodium	36 <del>JS</del>	64	5	mg/Kg	<del>JS</del>	03/19/05	SW846 3050B	SW846 6020
Thallium	0.20 <del>JS</del>	0.39	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Vanadium	32	0.39	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Zinc	1600	26	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	77.5	—	1	%		03/15/05	SM 2540G M	SM 2540G M

*me 04/05/05*

**En Chem****Analytical Report Number: 857072B**1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : NA-S2

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/31/05

Lab Sample Number : 857072-017

**INORGANICS**

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	8400	19	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Antimony	< 19 <i>uj</i>	19	10	mg/Kg	<i>g</i>	03/22/05	SW846 3050B	SW846 6010B
Arsenic	4.0	0.38	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Barium	120	0.38	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Beryllium	0.46	0.13	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cadmium	5.9	0.13	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Calcium	1100	38	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Chromium	11 <i>J</i>	0.38	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cobalt	4.2	0.25	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Copper	67	1.3	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Iron	9000	38	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Lead	120	0.31	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Magnesium	1000 <i>J</i>	19	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Manganese	260 <i>J</i>	0.25	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Mercury	0.031	0.013	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	11	0.38	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Potassium	730 <i>J</i>	25	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Selenium	0.88 <i>J</i>	<i>g</i> 1.3	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Silver	0.22	<i>g</i> 0.38	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Sodium	47	<i>g</i> 63	5	mg/Kg	<i>A</i>	03/19/05	SW846 3050B	SW846 6020
Thallium	0.17	<i>g</i> 0.38	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Vanadium	21	0.38	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Zinc	5100	25	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	79.9	—	1	%		03/15/05	SM 2540G M	SM 2540G M

*in 04/05/05*

# En Chem

## Analytical Report Number: 857072B

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : NA-S3

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/31/05

Lab Sample Number : 857072-018

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	11000	200	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Antimony	< 20 <b>43</b>	20	10	mg/Kg	<del>CN</del>	03/22/05	SW846 3050B	SW846 6010B
Arsenic	3.7	0.40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Barium	150	0.40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Beryllium	0.53	0.13	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cadmium	2.7	0.13	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Calcium	2300	40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Chromium	13 <b>J</b>	0.40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cobalt	3.7	0.26	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Copper	19	1.3	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Iron	11000	400	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Lead	40	0.33	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Magnesium	1200 <b>J</b>	20	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Manganese	260 <b>J</b>	0.26	5	mg/Kg	<del>/</del>	03/19/05	SW846 3050B	SW846 6020
Mercury	0.019	0.013	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	9.6	0.40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Potassium	870 <b>J</b>	26	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Selenium	0.59 <b>J</b> <del>P</del>	1.3	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Silver	0.11 <del>P</del>	0.40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Sodium	37 <del>P</del>	66	5	mg/Kg	<del>A</del>	03/19/05	SW846 3050B	SW846 6020
Thallium	0.16 <del>P</del>	0.40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Vanadium	28	0.40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Zinc	1500	26	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	75.8	—	1	%		03/15/05	SM 2540G M	SM 2540G M

see 04/05/05

# En Chem

## Analytical Report Number: 857072B

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : NA-S4

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/31/05

Lab Sample Number : 857072-019

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	7600	20	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Antimony	< 20 <b>UJ</b>	20	10	mg/Kg	<b>✓</b>	03/22/05	SW846 3050B	SW846 6010B
Arsenic	3.0	0.40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Barium	84	0.40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Beryllium	0.38	0.13	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cadmium	1.5	0.13	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Calcium	1700	40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Chromium	9.7 <b>J</b>	0.40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cobalt	2.9	0.27	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Copper	10	1.3	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Iron	7300	40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Lead	31	0.34	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Magnesium	920 <b>J</b>	20	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Manganese	280 <b>J</b>	0.27	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Mercury	0.015	0.013	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	6.6	0.40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Potassium	810 <b>J</b>	27	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Selenium	0.62 <b>J</b> <b>✓</b>	1.3	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Silver	0.10 <b>J</b> <b>✓</b>	0.40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Sodium	33 <b>✓</b>	67	5	mg/Kg	<b>✓</b>	03/19/05	SW846 3050B	SW846 6020
Thallium	0.13 <b>J</b> <b>✓</b>	0.40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Vanadium	19	0.40	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Zinc	950	27	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	74.5	—	1	%		03/15/05	SM 2540G M	SM 2540G M

*in 04/05/05*



**En Chem****Analytical Report Number: 857072B**1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : NA-S2D

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/31/05

Lab Sample Number : 857072-020

**INORGANICS**

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	8600	21	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Antimony	< 21 <b>uj</b>	21	10	mg/Kg	<b>C</b>	03/22/05	SW846 3050B	SW846 6010B
Arsenic	4.8	0.41	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Barium	93	0.41	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Beryllium	0.58	0.14	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cadmium	7.7	0.14	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Calcium	1500	41	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Chromium	13 <b>J</b>	0.41	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cobalt	6.6	0.28	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Copper	170	1.4	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Iron	10000	41	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Lead	230	0.35	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Magnesium	1100 <b>J</b>	21	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Manganese	320 <b>J</b>	0.28	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Mercury	0.050	0.014	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	37	0.41	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Potassium	750 <b>J</b>	28	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Selenium	1.1 <b>J</b>	1.4	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Silver	0.38 <b>J</b>	0.41	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Sodium	58 <b>J</b>	69	5	mg/Kg	<b>T</b>	03/19/05	SW846 3050B	SW846 6020
Thallium	0.17 <b>J</b>	0.41	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Vanadium	22	0.41	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Zinc	7700	28	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	72.4	---	1	%		03/15/05	SM 2540G M	SM 2540G M

*in 04/05/05*

# En Chem

A Division of Pace Analytical Services, Inc.

Analytical Report Number: 857072B

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : RR0-12D

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/31/05

Lab Sample Number : 857072-021

## INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	11000	17	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Antimony	< 17 <b>uJ</b>	17	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Arsenic	15	0.33	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Barium	420	0.33	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Beryllium	2.0	0.11	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cadmium	10	0.11	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Calcium	19000	33	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Chromium	38 <b>J</b>	0.33	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Cobalt	560	0.22	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Copper	3400	1.1	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Iron	73000	330	50	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Lead	520	0.28	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Magnesium	5200 <b>J</b>	17	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Manganese	1300 <b>J</b>	0.22	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Mercury	0.047	0.011	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	1100	0.33	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Potassium	1300 <b>J</b>	22	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Selenium	5.5	1.1	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Silver	34	0.33	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Sodium	1700	56	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Thallium	0.050 <b>J</b>	0.33	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Vanadium	20	0.33	5	mg/Kg		03/19/05	SW846 3050B	SW846 6020
Zinc	150000	220	100	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	90.0	—	1	%		03/15/05	SM 2540G M	SM 2540G M

in 04/05/05



**DATA VALIDATION**  
**FOR**  
**EAGLE ZINC PROJECT**

**INORGANICS ANALYSIS DATA**  
**Target Analyte List Metals in Soil Samples**  
**Samples Collected March 11, 2005**

**Chemical Analyses Performed by:**  
**EnChem (Division of Pace Analytical Services, Inc.)**  
**Green Bay, Wisconsin (All Analytes except Mercury)**  
**Kimberly, Wisconsin (Mercury)**  
**Sample Delivery Group Number 857072A**

**Data Deliverables Prepared for:**  
**ENVIRON International Corporation**  
**Deerfield, Illinois**

**Data Validation Performed by:**  
**Trillium, Inc.**  
**9312 Highland Gardens Road**  
**Baton Rouge, Louisiana 70811**  
**(225) 355-8702**  
**(225) 355-8987 (Fax)**

**April 4, 2005**

## EXECUTIVE SUMMARY

Validation of the inorganics analysis data [target analyte list (TAL) metals] was completed by Trillium, Inc. Analyses for mercury were performed by EnChem Laboratories in Kimberly, Wisconsin. Analyses for the remaining target analytes were performed by EnChem Laboratories in Green Bay, Wisconsin. The samples in this data set represent the sample collections from March 11, 2005, from the Eagle Zinc Project. Data for twelve soil samples were reported by the two laboratories in a single data package under Sample Delivery Group (SDG) 857072A, which was received for review on March 31, 2005. The data package includes the following samples:

---

RRO-12	NP-15	NP-16	RR2-11	RCO-10
CPH-9	NP-13	NP-14	CPH-6	RCO-5
RR1-4	RR1-3			

---

Findings of the validation effort resulted in the following qualifications of sample results:

- Results for cadmium in all of the samples in this data set were qualified as less than the reported values (U).
- Results for aluminum, arsenic, calcium, copper, magnesium, potassium, selenium, sodium, and thallium in all of the samples in this data set were qualified as estimated (UJ, J).
- The result for antimony in NP-16 was qualified as estimated (J).

Brief explanations of the reasons for the actions taken above may be found in the Overall Assessment (Section XII). Details of the validation findings and conclusions based on the review of the results for each quality control requirement are provided in the remaining sections of this report.

The validator removed all "A," "B," "C," "E," "N," "&," and "\*" qualifiers applied by the laboratory.

Chain of custody documentation issues are discussed in Section XI.

This validation report should be considered part of the data package for all future distributions of the inorganics data.



## INTRODUCTION

Analyses were performed according to EPA Methods 3050B, 7471A, 6010B, and 6020 (SW-846; Test Methods for Evaluating Solid Waste, Third Edition, through Update III, 12/96). Results of sample analyses are reported by the laboratory as either qualified or unqualified; various qualifier codes are used by the laboratory to denote specific information regarding the analytical results. In this data set, the laboratory used the following defined qualifiers:

- A Analyte is detected in the method blank. Method blank criteria are evaluated to the laboratory method detection limit. Additionally, method blank acceptance may be based on project specific criteria or determined from analyte concentrations in the sample and are evaluated on a sample by sample basis.
- B The analyte has been detected between the method detection limit and the reporting limit.
- C Elevated detection limit.
- E Estimated concentration due to matrix interferences. During the metals analysis the serial dilution failed to meet the established control limits of 0-10%. The sample concentration is greater than 50 times the IDL for analysis done on the ICP or 100 times the IDL for analysis done on the ICP-MS. The result was flagged with the E qualifier to indicate that a physical interference was observed.
- N Spiked sample recovery not within control limits.
- & Laboratory Control Spike recovery not within control limits.
- \* Precision not within control limits.

The validator removed all "A," "B," "C," "E," "N," "&," and "\*" qualifiers applied by the laboratory.

To the extent applicable, Trillium's validation was performed in conformance with the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (EPA 540-R-01-008, July 2002). Where discrepancies were found, the specifications of the referenced methods took precedence. In addition, professional judgment was applied as necessary and appropriate.

The data validation process is intended to evaluate data on a technical basis rather than a contract compliance basis. This requires that the data package contain sufficient raw data documentation to facilitate the validation process and allow verification of all reported results. It is also assumed that the data package represents the best efforts of the laboratory and has already been subjected to adequate and sufficient quality review prior to submission for validation.

During the validation process, laboratory data are verified against all available supporting documentation. Based on this evaluation, qualifier codes may be added, deleted, or modified by the data validator. Validated results are, therefore, either qualified or unqualified. Unqualified results mean that the reported values may be used without reservation. Final validated results are annotated with the following codes as defined in the National Functional Guidelines:

- U      The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
- J      The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
- J+    The result is an estimated quantity, but the result may be biased high.
- J-    The result is an estimated quantity, but the result may be biased low.
- R      The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.
- UJ    The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

These codes are recorded on the Data Summary Forms contained in Attachment A, as well as on the laboratory answer forms in Attachment B of this validation report to qualify the results as appropriate according to the review of the data package.

Two facts should be noted by all data users. First, **the “R” qualifier means that the laboratory-reported value is unusable.** In other words, due to significant quality control problems, the analysis is invalid and provides no information as to whether the compound is present or not. Rejected values should not appear on data tables because they cannot be relied upon, even as a last resort. Second, **no analyte concentration is guaranteed to be accurate even if all associated quality control is acceptable.** Strict quality control conformance serves only to increase confidence in reported results; any analytical result will always contain some error.

The data user is also cautioned that the validation effort is based on the raw data printouts as provided by the laboratory. Software manipulation cannot be routinely detected during validation; unless otherwise stated in the report, these kinds of issues are outside the scope of this review.

## **I. Holding Times, Preservation and Sample Integrity**

The soil samples were collected on March 11, 2005. All of the samples were digested and analyzed well within acceptable holding times (28 days for mercury and 180 days for the remaining analytes).

The temperature of the sample cooler (1°C), as recorded on the Chain of Custody Record and the Cooler Receipt Log, was slightly below the acceptable temperature range (4°C  $\pm$  2°C). Based on professional judgment, no adverse effect on the metals results would be expected based on the slightly low cooler temperature and no data were qualified on this basis.

The Chain of Custody Record and Cooler Receipt Log also indicated that the samples were received intact but no custody seals were used on the sample cooler.

Copies of the FedEx airbills were provided by the laboratory on April 4, 2005, to document custody of the samples between the field and the laboratory.

## **II. Mass Spectrometer Tune**

The tuning solution for the ICP-MS was analyzed on March 18, 2005 at 05:30. All criteria specified in Method 6020 were met.

As required by the analytical method, a blank and three calibration standards were analyzed to document instrument stability. The laboratory confirmed that three integrations of each target analyte were performed but only the average of these integrations was reported in the supporting data. Percent relative standard deviation (%RSD) values were reported by the laboratory but could not be confirmed by the validator since abbreviated raw data were provided. No acceptance limits are provided in Method 6020 but all reported %RSD values were less than 10%, which was deemed acceptable based on professional judgment.

## **III. Calibrations**

Inductively coupled plasma-mass spectrometry (ICP-MS) analyses were performed on an instrument identified as "HP ICPMS 2." The samples reported in this data set were analyzed in two analysis series on March 18 and 21, 2005.

Inductively coupled plasma-atomic emission spectroscopy (ICP-AES) analyses were performed on a trace ICP-AES instrument identified as "TJA61E Trace ICP 3." The samples reported in this data set were analyzed in two analysis series on March 22 and 23, 2005.

Mercury analyses were performed in a single analysis series run on March 17, 2005 on a cold vapor atomic absorption (CVAA) system identified as "PE CVAA 1."



Initial calibration verification (ICV) standards were performed for all of the target analytes after each initial calibration. All ICV percent recoveries (%R) were correctly calculated, accurately reported, and within the validation guideline-specified control limits (90-110% for analytes determined by ICP-MS and ICP-AES and 80-120% for mercury).

Continuing calibration verification (CCV) standards were run at the appropriate frequency (after every ten samples) in all of the analysis series. All %Rs were correctly calculated, accurately reported, and within the method- and validation guideline-specified control limits (90-110% for analytes determined by ICP-MS and ICP-AES and 80-120% for mercury).

A linearity check for mercury was performed at the start of the analysis series and gave an acceptable correlation coefficient ( $>0.995$ ).

The analysis of "CRI" standards in the ICP-MS and ICP-AES analyses and "CRA" standards in the mercury analyses are not required by SW-846 methods and were not performed in association with the reported analyses.

#### **IV. Blanks**

Initial and continuing calibration blanks (ICBs and CCBs) were analyzed at the appropriate frequencies throughout each analytical sequence. Copper ( $1.20\ \mu\text{g/L}$  to  $3.71\ \mu\text{g/L}$ ), lead ( $0.34\ \mu\text{g/L}$  to  $0.94\ \mu\text{g/L}$ ), and nickel ( $0.34\ \mu\text{g/L}$  to  $1.11\ \mu\text{g/L}$ ) were reported in three of the four CCBs and manganese ( $0.20\ \mu\text{g/L}$ ) was reported in one of the CCBs. With regard to ICBs and CCBs, the validation guidance document does not include a multiplier of the concentration detected in these blanks for use in assessing blank contamination. Therefore, the absolute concentrations in the CCBs noted above were used for evaluation purposes. Results for copper, lead, nickel, and manganese were significantly greater than the concentrations of these analytes detected in the associated CCBs (i.e., all were greater than five times the highest concentration detected in any of the CCBs); therefore, no data were qualified on this basis.

Aluminum ( $12.33\ \text{mg/kg}$ ), cadmium ( $36.32\ \text{mg/kg}$ ), copper ( $0.795\ \text{mg/kg}$ ), iron ( $31.32\ \text{mg/kg}$ ), lead ( $0.20\ \text{mg/kg}$ ), magnesium ( $11.0\ \text{mg/kg}$ ), manganese ( $0.245\ \text{mg/kg}$ ), nickel ( $0.280\ \text{mg/kg}$ ), potassium ( $12.2\ \text{mg/kg}$ ), and sodium ( $27.545\ \text{mg/kg}$ ) were detected in the solid-matrix preparation blank (PB). With regard to PBs, the validation guidance document indicates that if an analyte is detected in the PB below the contract required quantitation limit (CRQL), "no correction of the sample results should be performed." For these samples, the estimated quantitation limit (EQL) was substituted for the CRQL. Only cadmium was detected above the EQL, therefore, results for cadmium in all of the samples in this data set were qualified as less than the reported values (U) based on detection in the samples at concentrations less than that detected in the PB. Results for aluminum, copper, iron, lead, magnesium, manganese, nickel, potassium and sodium were significantly greater than the concentrations detected in the PB (i.e., all were greater than five times the concentration detected in the PB); therefore, results for these analytes were not qualified on this basis.



No field-submitted blanks were included in this data set.

## V. ICP Interference Check Sample

ICP interference check sample analyses were performed at the appropriate frequency throughout all analysis series. Target analyte recoveries were acceptable (80-120%) in all cases.

## VI Spiked Sample Analyses

### A. Laboratory Control Samples

A solid-matrix laboratory control sample pair (LCS/LCSD) was prepared and analyzed with each analysis series. Each LCS/LCSD pair included all of the target analytes required for that analytical sequence. All percent recovery (%R) and relative percent difference (RPD) values were correctly calculated, accurately reported, and within the analyte-specific acceptance limits specified by the laboratory on the summary forms.

### B. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analyses

RR2-11 was prepared as an MS/MSD pair for all of the target analytes. All %R and RPD values were correctly calculated and accurately reported. For the target analytes with an amount of spike added greater than four times the concentration of the analyte in the original analysis of RR2-11, %Rs and RPDs were within the analyte-specific acceptance limits specified by the laboratory on the summary forms with the following exceptions:

Analyte	MS	MSD	Acceptance Limits
arsenic	71%		75-125%
calcium	28%		75-125%
selenium	70%	71%	78-125%
sodium	66%	73%	75-125%
thallium	65%	57%	75-125%

Based on unacceptably low recoveries in one or both of the matrix spike analyses, results for arsenic, calcium, selenium, sodium, and thallium were qualified as estimated (J).

For the target analytes with an amount of spike added less than four times the concentration of the analyte detected in the original analysis of RR2-11, acceptable recoveries would not be expected. Since the amount of spike added is insignificant, this presents an opportunity to compare precision among the triplicate analyses (sample, MS, and MSD). Based on professional judgment, an acceptance limit of  $\leq 25\%$  RSD was used to evaluate the precision among results reported for aluminum, barium, chromium, iron, lead, manganese, nickel, cobalt, silver, antimony, and zinc. Acceptable precision was observed (2.8% to 12.0%) in all cases.



Post digestion spike recoveries for all of the TAL target analytes were acceptable (75-125% acceptance limits).

## **VII. Duplicate Sample Analyses**

### **A. Laboratory Duplicate Analysis**

No laboratory duplicate analysis was performed on a sample in this data set.

### **B. Field Duplicate Analyses**

No field duplicate pair was identified in this sample set.

## **VIII Internal Standard Performance**

Lithium (mass 6), scandium (mass 45), germanium (mass 74), yttrium (mass 89), indium (mass 115), terbium (mass 159), and bismuth (mass 209) were included as internal standard analytes for the ICP-MS analyses. Percent recoveries of the internal standards were within the analytical method-specified 30-120% acceptance limits in all of the site and quality control sample analyses.

## **IX ICP Serial Dilution**

ICP-AES and ICP-MS serial dilution analyses were performed on RRO-12. Results for elements with initial (undiluted) results greater than 10 times the instrument detection limit for analytes determined by ICP-AES and greater than 100 times the concentration of the analyte in the reagent blank for analytes determined by ICP-MS were acceptable (<10% Difference) with the exceptions of aluminum (12%), copper (12%), magnesium (16%), potassium (13%), and sodium (11%). Based on unacceptable results in the serial dilution analyses, results for aluminum, copper, magnesium, potassium, and sodium in all of the soil samples in this data set were qualified as estimated (J).

## **X. Sample Results Verification**

The laboratory confirmed that three integrations of each target analyte were performed but only the average of these integrations was reported in the supporting data; therefore, the reported average concentration could not be confirmed by the validator. Based on the reported average concentrations, all sample results were correctly calculated and accurately reported from the raw data, including adjustments for the solid-matrix preparation procedures, dilutions, and percent solids.



Instrument detection limits (IDLs) were reported for all three instruments used to perform the reported sample results. IDLs were established on April 4, 2004, on the ICP-MS instrument, on January 5, 2004, on the ICP-AES instrument, and on July 5, 2004, on the instrument used to perform the mercury analyses. The analytical methods specify that IDLs must be established within three months of the associated sample analyses.

Linear ranges were established on October 4, 2004, on the instrument used to perform ICP-MS analyses and on January 27, 2005, on the instrument used to perform the ICP-AES analyses. Method 6020 does not specify a required frequency for the establishment of linear ranges, but Method 6010B specifies that linear ranges must be established within three months of the associated sample analyses.

Interelement correction factors were performed on December 31, 2003, on the instrument used to perform ICP-AES analyses. Method 6010B specifies that interelement correction factors must be established within one year of the associated sample analyses. Interelement correction factors are not required for analyses performed by ICP-MS.

Based on professional judgment, as concentrations approach the IDL the accuracy of the measurement decreases; values closer to the EQL, however, are probably quite accurate. Therefore, a guideline of two times the IDL was used to determine whether the reported results warranted qualification. Sample results below the respective reporting limit, less than two times the IDL, and not otherwise qualified were qualified as estimated (J). Specifically, the result for *antimony in NP-16* was so qualified.

## **XI. Documentation**

Chain of custody records were present and accurately completed for all samples reported in this data package with the following exceptions:

- The chain of custody record indicates these samples were “relinquished by” Ross Jones on March 12, 2005, but does not indicate a “received by” signature, date, or time. A “relinquished by” FedEx is indicated on March 14, 2005, and a laboratory “received by” signature indicates the samples were received on March 14, 2005, at 09:20. A copy of the FedEx Airbill was requested, which indicated the samples were shipped on March 12, 2005, were in transit with FedEx on March 13, 2005, and were delivered on March 14, 2005. A copy of the airbill should be included in the original and all copies of the data package to ensure that complete documentation is available for future reference.
- The chain of custody record and laboratory cooler receipt log indicate that no custody seals were used on the sample cooler.

No data presentation problems were noted in the data package received for review.



These chain of custody issues do not directly affect the validity of the reported results, but they could be problematic if these data are used in litigation.

## **XII. Overall Assessment**

Findings of the validation effort resulted in the following qualifications of sample results:

- Results for cadmium in all of the samples in this data set were qualified as less than the reported values (U) due to detection of this analyte at concentrations less than the concentration detected in the solid-matrix preparation blank.
- Based on unacceptably low recoveries in one or both of the matrix spike analyses, results for arsenic, calcium, selenium, sodium, and thallium in all of the samples in this data set were qualified as estimated (UJ, J). Results for sodium in these samples were also estimated due to an unacceptable result in the serial dilution analysis.
- Based on unacceptable results in the serial dilution analyses, results for aluminum, copper, magnesium, and potassium in all of the soil samples in this data set were qualified as estimated (J).
- Based on professional judgment, the result for antimony in NP-16 was qualified as estimated (J) because the reported concentration was less than two times the instrument detection limit and not otherwise qualified.

Chain of custody documentation issues are discussed in Section XI.

This validation report should be considered part of the data package for all future distributions of the inorganics data.



**ATTACHMENT A**

**DATA SUMMARY FORMS**

**Target Analyte List Metals in Soil Samples  
Sample Delivery Group Number 857072A**

DATA SUMMARY FORM: INORGANICS  
SOIL SAMPLES  
(mg/kg)

Site Name: Eagle Zinc

Sampling Date: March 11, 2005

Laboratory Sample Delivery Group Number: 857072A

Trillium Project No.: 05906

Sample Number Lab ID Percent Solids		RRO-12 857072-001 80.0	NP-15 857072-002 90.2	NP-16 857072-003 78.3	RR2-11 857072-004 93.9	RCO-10 857072-005 94.1	CPH-9 857072-006 94.2	NP-13 857072-007 89.2	NP-14 857072-008 93.2
EQL	Analyte								
15	Aluminum	7700 J	9600 J	6000 J	35000 J	20000 J	3800 J	8300 J	3900 J
7.6	Antimony	41	110	3.8 J	400	190	16 U	17 U	16 U
0.30	Arsenic	11 J	11 J	12 J	21 J	41 J	8.1 J	5.7	3.1 J
0.30	Barium	170	110	130	130	350	150	290	210
0.10	Beryllium	1.6	0.97	0.86	1.5	2.4	0.68	1.2	0.66
0.10	Cadmium	6.9 U	19 U	15 U	7.2 U	24 U	6.1 U	23 U	32 U
30	Calcium	17000 J	8200 J	16000 J	3300 J	20000 J	7500 J	5000 J	1900 J
0.30	Chromium	47	62	22	290	220	4.4	11	4.9
0.20	Cobalt	440	500	430	93	760	440	8.2	4.4
1.0	Copper	2200 J	1900 J	1900 J	34000 J	24000 J	2100 J	190 J	140 J
30	Iron	48000	31000	36000	77000	60000	47000	24000	5500
0.25	Lead	810	1200	550	7700	2500	79	76	74
15	Magnesium	4700 J	3000 J	3800 J	1200 J	5400 J	4400 J	700 J	570 J
0.20	Manganese	930	510	1100	750	880	330	490	65
0.100	Mercury	0.090	0.100	0.230	0.012	0.024	0.046	0.028	0.036
0.30	Nickel	1000	1300	800	10000	7000	610	21	10
20	Potassium	700 J	410 J	640 J	230 J	1400 J	770 J	600 J	240 J
1.0	Selenium	4.0 J	8.1 J	5.7 J	3.6 J	4.8 J	4.4 J	1.8 J	2.8 J
0.30	Silver	18	9.5	21	29	43	48	0.39	0.48
50	Sodium	1100 J	170 J	1100 J	250 J	810 J	450 J	460 J	220 J
0.30	Thallium	0.11 J	0.12 J	0.11 J	1.0 J	0.09 J	0.32 UJ	0.24 J	0.07 J
0.30	Vanadium	17	9.8	18	5.7	14	12	29	12
10	Zinc	120000	180000	150000	140000	130000	170000	25000	39000

DATA SUMMARY FORM: INORGANICS  
SOIL SAMPLES  
(mg/kg)

Site Name: Eagle Zinc

Sampling Date: March 11, 2005

Laboratory Sample Delivery Group Number: 857072A

Trillium Project No.: 05906

Sample Number		CPH-6	RCO-5	RR1-4	RR1-3				
Lab ID		857072-009	857072-010	857072-011	857072-012				
Percent Solids		95.7	92.0	95.1	91.8				
EQL	Analyte								
15	Aluminum	7000 J	8300 J	6000 J	4500 J				
7.6	Antimony	8.3	6.5	16 U	16 U				
0.30	Arsenic	33 J	19 J	7.9 J	16 J				
0.30	Barium	210	230	150	480				
0.10	Beryllium	1.3	2.9	0.89	0.86				
0.10	Cadmium	10 U	21 U	4.9 U	35 U				
30	Calcium	9900 J	17000 J	9400 J	950 J				
0.30	Chromium	10	30	6.8	12				
0.20	Cobalt	250	570	880	9.7				
1.0	Copper	2400 J	2200 J	2600 J	400 J				
30	Iron	110000	25000	72000	88000				
0.25	Lead	500	530	120	1600				
15	Magnesium	4200 J	3800 J	6000 J	340 J				
0.20	Manganese	910	570	290	160				
0.100	Mercury	0.430	0.056	0.038	0.075				
0.30	Nickel	650	1100	890	22				
20	Potassium	1300 J	470 J	630 J	340 J				
1.0	Selenium	6.9 J	5.8 J	3.5 J	1.7 J				
0.30	Silver	14	13	77	1.8				
50	Sodium	340 J	730 J	340 J	130 J				
0.30	Thallium	0.31 UJ	0.10 J	0.32 UJ	0.10 J				
0.30	Vanadium	11	15	10	27				
10	Zinc	190000	200000	130000	7700				



**ATTACHMENT B**

**LABORATORY ANSWER FORMS**

**Target Analyte List Metals in Soil Samples  
Sample Delivery Group Number 857072A**



**En Chem****Analytical Report Number: 857072A**1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : RRO-12

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/29/05

Lab Sample Number : 857072-001

**INORGANICS**

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	7700 J	19	5	mg/Kg	<del>F</del>	03/18/05	SW846 3050B	SW846 6020
Antimony	41	19	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Arsenic	11 J	0.37	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Barium	170	0.37	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Beryllium	1.6	0.12	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cadmium	6.9 u	0.12	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Calcium	17000 J	37	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Chromium	47	0.37	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cobalt	440	0.25	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Copper	2200 J	1.2	5	mg/Kg	<del>P</del>	03/18/05	SW846 3050B	SW846 6020
Iron	48000	37	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Lead	810	0.31	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Magnesium	4700 J	19	5	mg/Kg	<del>F</del>	03/18/05	SW846 3050B	SW846 6020
Manganese	930	0.25	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Mercury	0.090	0.012	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	1000	0.37	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Potassium	700 J	25	5	mg/Kg	<del>P</del>	03/18/05	SW846 3050B	SW846 6020
Selenium	4.0 J	1.2	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Silver	18	0.37	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Sodium	1100 J	62	5	mg/Kg	<del>P</del>	03/18/05	SW846 3050B	SW846 6020
Thallium	0.11 J <del>P</del>	0.37	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Vanadium	17	0.37	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Zinc	120000	250	100	mg/Kg	<del>P</del>	03/22/05	SW846 3050B	SW846 6010B
Percent Solids	80.0	---	1	%		03/15/05	SM 2540G M	SM 2540G M

en 04/04/05

# En Chem

A Division of Pace Analytical Services, Inc.

Analytical Report Number: 857072A

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : NP-15

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/29/05

Lab Sample Number : 857072-002

## INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	9600 J	17	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Antimony	110	17	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Arsenic	11 J	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Barium	110	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Beryllium	0.97	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cadmium	19 u	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Calcium	8200 J	33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Chromium	62	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cobalt	500	0.22	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Copper	1900 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Iron	31000	33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Lead	1200	0.28	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Magnesium	3000 J	17	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Manganese	510	0.22	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Mercury	0.10	0.011	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	1300	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Potassium	410 J	22	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Selenium	8.1 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Silver	9.5	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Sodium	170 J	55	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Thallium	0.12 J	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Vanadium	9.8	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Zinc	180000	220	100	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	90.2	--	1	%		03/15/05	SM 2540G M	SM 2540G M

in 04/04/05

# En Chem

## Analytical Report Number: 857072A

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : NP-16

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/29/05

Lab Sample Number : 857072-003

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	6000 J	19	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Antimony	3.8 J <del>8</del>	19	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Arsenic	12 J	0.38	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Barium	130	0.38	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Beryllium	0.86	0.13	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cadmium	15 u	0.13	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Calcium	16000 J	38	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Chromium	22	0.38	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cobalt	430	0.25	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Copper	1900 J	1.3	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Iron	36000	38	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Lead	550	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Magnesium	3800 J	19	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Manganese	1100	0.25	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Mercury	0.23	0.013	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	800	0.38	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Potassium	640 J	25	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Selenium	5.7 J	1.3	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Silver	21	0.38	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Sodium	1100 J	64	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Thallium	0.11 J <del>8</del>	0.38	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Vanadium	18	0.38	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Zinc	150000	250	100	mg/Kg	<del>8</del>	03/22/05	SW846 3050B	SW846 6010B
Percent Solids	78.3	—	1	%		03/15/05	SM 2540G M	SM 2540G M

see 04/04/05

# En Chem

## Analytical Report Number: 857072A

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : RR2-11

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/29/05

Lab Sample Number : 857072-004

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	35000 J	1600	500	mg/Kg		03/21/05	SW846 3050B	SW846 6020
Antimony	400	16	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Arsenic	21 J	0.32	5	mg/Kg	<del>N</del>	03/18/05	SW846 3050B	SW846 6020
Barium	130	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Beryllium	1.5	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cadmium	7.2 u	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Calcium	3300 J	32	5	mg/Kg	<del>N</del>	03/18/05	SW846 3050B	SW846 6020
Chromium	290	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cobalt	93	0.21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Copper	34000 J	110	500	mg/Kg		03/21/05	SW846 3050B	SW846 6020
Iron	77000	3200	500	mg/Kg		03/21/05	SW846 3050B	SW846 6020
Lead	7700	27	500	mg/Kg	<del>N</del>	03/21/05	SW846 3050B	SW846 6020
Magnesium	1200 J	16	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Manganese	750	2.1	50	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Mercury	0.012	0.011	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	10000	32	500	mg/Kg		03/21/05	SW846 3050B	SW846 6020
Potassium	230 J	21	5	mg/Kg	<del>A</del>	03/18/05	SW846 3050B	SW846 6020
Selenium	3.6 J	1.1	5	mg/Kg	<del>N</del>	03/18/05	SW846 3050B	SW846 6020
Silver	29	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Sodium	250 J	53	5	mg/Kg	<del>N</del>	03/18/05	SW846 3050B	SW846 6020
Thallium	1.0 J <del>N</del>	3.2	50	mg/Kg	<del>N</del>	03/18/05	SW846 3050B	SW846 6020
Vanadium	5.7	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Zinc	140000	2100	1000	mg/Kg	<del>N</del>	03/23/05	SW846 3050B	SW846 6010B
Percent Solids	93.9	---	1	%		03/15/05	SM 2540G M	SM 2540G M

ew 04/04/05

# En Chem

## Analytical Report Number: 857072A

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : RCO-10

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/29/05

Lab Sample Number : 857072-005

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	20000 J	160	50	mg/Kg		03/21/05	SW846 3050B	SW846 6020
Antimony	190	16	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Arsenic	41 J	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Barium	350	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Beryllium	2.4	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cadmium	24 u	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Calcium	20000 J	32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Chromium	220	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cobalt	760	0.21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Copper	24000 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Iron	60000	320	50	mg/Kg		03/21/05	SW846 3050B	SW846 6020
Lead	2500	0.27	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Magnesium	5400 J	16	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Manganese	880	0.21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Mercury	0.024	0.011	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	7000	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Potassium	1400 J	21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Selenium	4.8 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Silver	43	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Sodium	810 J	53	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Thallium	0.085 J	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Vanadium	14	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Zinc	130000	210	100	mg/Kg	8	03/22/05	SW846 3050B	SW846 6010B
Percent Solids	94.1	—	1	%		03/15/05	SM 2540G M	SM 2540G M

u 04/04/05

# En Chem

## Analytical Report Number: 857072A

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : CPH-9

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/29/05

Lab Sample Number : 857072-006

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	3800 J	16	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Antimony	< 16	16	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Arsenic	8.1 J	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Barium	150	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Beryllium	0.68	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cadmium	6.1 u	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Calcium	7500 J	32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Chromium	4.4	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cobalt	440	0.21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Copper	2100 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Iron	47000	32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Lead	79	0.27	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Magnesium	4400 J	16	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Manganese	330	0.21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Mercury	0.046	0.011	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	610	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Potassium	770 J	21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Selenium	4.4 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Silver	48	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Sodium	450 J	53	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Thallium	< 0.32 uJ	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Vanadium	12	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Zinc	170000	210	100	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	94.2	---	1	%		03/15/05	SM 2540G M	SM 2540G M

see 04/04/05

# En Chem

## Analytical Report Number: 857072A

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : NP-13

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/29/05

Lab Sample Number : 857072-007

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	8300 J	17	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Antimony	< 17	17	10	mg/Kg	✓	03/22/05	SW846 3050B	SW846 6010B
Arsenic	5.7 J	0.34	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Barium	290	0.34	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Beryllium	1.2	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cadmium	23 u	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Calcium	5000 J	34	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Chromium	11	0.34	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cobalt	8.2	0.22	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Copper	190 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Iron	24000	34	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Lead	76	0.28	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Magnesium	700 J	17	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Manganese	490	0.22	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Mercury	0.028	0.011	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	21	0.34	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Potassium	600 J	22	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Selenium	1.8 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Silver	0.39	0.34	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Sodium	460 J	56	5	mg/Kg	✓	03/18/05	SW846 3050B	SW846 6020
Thallium	0.24 J	0.34	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Vanadium	29	0.34	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Zinc	25000	23	10	mg/Kg	✓	03/22/05	SW846 3050B	SW846 6010B
Percent Solids	89.2	--	1	%		03/15/05	SM 2540G M	SM 2540G M

see 04/04/05

# En Chem

## Analytical Report Number: 857072A

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : NP-14

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/29/05

Lab Sample Number : 857072-008

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	3900 J	16	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Antimony	< 16	16	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Arsenic	3.1 J	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Barium	210	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Beryllium	0.66	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cadmium	32 u	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Calcium	1900 J	32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Chromium	4.9	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cobalt	4.4	0.21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Copper	140 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Iron	5500	32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Lead	74	0.27	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Magnesium	570 J	16	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Manganese	65	0.21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Mercury	0.036	0.011	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	10	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Potassium	240 J	21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Selenium	2.8 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Silver	0.48	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Sodium	220 J	54	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Thallium	0.070 J	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Vanadium	12	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Zinc	39000	21	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	93.2	—	1	%		03/15/05	SM 2540G M	SM 2540G M

see 04/04/05



**En Chem****Analytical Report Number: 857072A**1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Face Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : CPH-6

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/29/05

Lab Sample Number : 857072-009

**INORGANICS**

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	7000 J	16	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Antimony	8.3 B	16	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Arsenic	33 J	0.31	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Barium	210	0.31	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Beryllium	1.3	0.10	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cadmium	10 u	0.10	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Calcium	9900 J	31	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Chromium	10	0.31	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cobalt	250	0.21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Copper	2400 J	1.0	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Iron	110000	310	50	mg/Kg		03/21/05	SW846 3050B	SW846 6020
Lead	800	0.26	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Magnesium	4200 J	16	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Manganese	910	0.21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Mercury	0.43	0.010	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	650	0.31	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Potassium	1300 J	21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Selenium	6.9 J	1.0	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Silver	14	0.31	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Sodium	340 J	52	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Thallium	< 0.31 u	0.31	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Vanadium	11	0.31	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Zinc	190000	210	100	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	95.7	—	1	%		03/15/05	SM 2540G M	SM 2540G M

see 04/04/05

# En Chem

## Analytical Report Number: 857072A

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Face Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : RCO-5

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/29/05

Lab Sample Number : 857072-010

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	8300 J	16	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Antimony	6.5	16	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Arsenic	19 J	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Barium	230	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Beryllium	2.9	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cadmium	21 u	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Calcium	17000 J	33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Chromium	30	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cobalt	570	0.22	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Copper	2200 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Iron	25000	33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Lead	530	0.27	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Magnesium	3800 J	16	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Manganese	570	0.22	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Mercury	0.056	0.011	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	1100	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Potassium	470 J	22	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Selenium	5.8 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Silver	13	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Sodium	730 J	54	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Thallium	0.098 J	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Vanadium	15	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Zinc	200000	220	100	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	92.0	---	1	%		03/15/05	SM 2540G M	SM 2540G M

see 04/04/05

# En Chem

## Analytical Report Number: 857072A

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : RR1-4

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/29/05

Lab Sample Number : 857072-011

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	6000 J	16	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Antimony	< 16	16	10	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Arsenic	7.9 J	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Barium	150	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Beryllium	0.89	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cadmium	4.9 u	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Calcium	9400 J	32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Chromium	6.8	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cobalt	880	0.21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Copper	2600 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Iron	72000	320	50	mg/Kg		03/21/05	SW846 3050B	SW846 6020
Lead	120	0.26	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Magnesium	6000 J	16	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Manganese	290	0.21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Mercury	0.038	0.011	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	890	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Potassium	630 J	21	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Selenium	3.5 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Silver	77	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Sodium	340 J	53	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Thallium	< 0.32 uJ	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Vanadium	10	0.32	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Zinc	130000	210	100	mg/Kg		03/22/05	SW846 3050B	SW846 6010B
Percent Solids	95.1	--	1	%		03/15/05	SM 2540G M	SM 2540G M

see 04/04/05

**En Chem****Analytical Report Number: 857072A**1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number : 21-7400E

Field ID : RR1-3

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/29/05

Lab Sample Number : 857072-012

**INORGANICS**

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	4500 J	16	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Antimony	< 16	16	10	mg/Kg	<del>C</del>	03/22/05	SW846 3050B	SW846 6010B
Arsenic	16 J	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Barium	480	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Beryllium	0.86	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cadmium	35 u	0.11	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Calcium	950 J	33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Chromium	12	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Cobalt	9.7	0.22	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Copper	400 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Iron	88000	330	50	mg/Kg		03/21/05	SW846 3050B	SW846 6020
Lead	1600	0.27	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Magnesium	340 J	16	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Manganese	160	0.22	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Mercury	0.075	0.011	1	mg/kg		03/17/05	SW846 7471A	SW846 7471A
Nickel	22	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Potassium	340 J	22	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Selenium	1.7 J	1.1	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Silver	1.8	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Sodium	130 J	54	5	mg/Kg	<del>A</del>	03/18/05	SW846 3050B	SW846 6020
Thallium	0.098 J	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Vanadium	27	0.33	5	mg/Kg		03/18/05	SW846 3050B	SW846 6020
Zinc	7700	22	10	mg/Kg	<del>A</del>	03/22/05	SW846 3050B	SW846 6010B
Percent Solids	91.8	---	1	%		03/15/05	SM 2540G M	SM 2540G M

en 04/04/05



**DATA VALIDATION**

**FOR**

**EAGLE ZINC PROJECT**

**INORGANICS ANALYSIS DATA**

**Target Analyte List Metals in A Composite Soil Sample  
Samples for Compositing Collected March 11, 2005**

**Chemical Analyses Performed by:**

**EnChem (Division of Pace Analytical Services, Inc.)  
Green Bay, Wisconsin (All Analytes except Mercury)  
Kimberly, Wisconsin (Mercury)  
Sample Delivery Group Number 857319**

**Data Deliverables Prepared for:  
ENVIRON International Corporation  
Deerfield, Illinois**

**Data Validation Performed by:  
Trillium, Inc.  
9312 Highland Gardens Road  
Baton Rouge, Louisiana 70811  
(225) 355-8702  
(225) 355-8987 (Fax)**

**April 5, 2005**



## EXECUTIVE SUMMARY

Validation of the inorganics analysis data [target analyte list (TAL) metals] was completed by Trillium, Inc. Analyses for mercury were performed by EnChem Laboratories in Kimberly, Wisconsin. Analyses for the remaining target analytes were performed by EnChem Laboratories in Green Bay, Wisconsin. The sample in this data set represents a composite of fifteen soil samples collected on March 11, 2005, from the Eagle Zinc Project. Data for the composite soil sample were reported by the two laboratories in a single data package under Sample Delivery Group (SDG) 857319, which was received for review on April 1, 2005. After compositing by the laboratory, the sample in this data package was identified as "Composite Sample." The Composite Sample included the following samples:

---

RRO-12F	NP-15F	NP-16F	RR-2-11F	RCO-10F	CPH-9F
NP-13F	NP-14F	CPH-6F	RCO-5F	RR1-4F	RR1-3F
RR1-2F	RR1-1F	MP1-21F			

---

Findings of the validation effort resulted in the following qualifications of sample results:

- The result for beryllium in Composite Sample was qualified as J-; this result may be biased low.
- The result for antimony in Composite Sample was rejected (R).

Brief explanations of the reasons for the actions taken above may be found in the Overall Assessment (Section XII). Details of the validation findings and conclusions based on the review of the results for each quality control requirement are provided in the remaining sections of this report.

The validator removed all "B," "C," "N," and "\*" qualifiers applied by the laboratory.

Chain of custody documentation issues are discussed in Section XI.

This validation report should be considered part of the data package for all future distributions of the inorganics data.



## INTRODUCTION

Analyses were performed according to EPA Methods 3050B, 7471A, and 6010B (SW-846; Test Methods for Evaluating Solid Waste, Third Edition, through Update III, 12/96). Results of sample analyses are reported by the laboratory as either qualified or unqualified; various qualifier codes are used by the laboratory to denote specific information regarding the analytical results. In this data set, the laboratory used the following defined qualifiers:

- B The analyte has been detected between the method detection limit and the reporting limit.
- C Elevated detection limit.
- N Spiked sample recovery not within control limits.
- \* Precision not within control limits.

The validator removed all "B," "C," "N," and "\*" qualifiers applied by the laboratory.

To the extent applicable, Trillium's validation was performed in conformance with the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (EPA 540-R-01-008, July 2002). Where discrepancies were found, the specifications of the referenced methods took precedence. In addition, professional judgment was applied as necessary and appropriate.

The data validation process is intended to evaluate data on a technical basis rather than a contract compliance basis. This requires that the data package contain sufficient raw data documentation to facilitate the validation process and allow verification of all reported results. It is also assumed that the data package represents the best efforts of the laboratory and has already been subjected to adequate and sufficient quality review prior to submission for validation.

During the validation process, laboratory data are verified against all available supporting documentation. Based on this evaluation, qualifier codes may be added, deleted, or modified by the data validator. Validated results are, therefore, either qualified or unqualified. Unqualified results mean that the reported values may be used without reservation. Final validated results are annotated with the following codes as defined in the National Functional Guidelines:

- U The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
- J The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
- J+ The result is an estimated quantity, but the result may be biased high.

- J- The result is an estimated quantity, but the result may be biased low.
- R The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.
- UJ The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

These codes are recorded on the Data Summary Form contained in Attachment A, as well as on the laboratory answer forms in Attachment B of this validation report to qualify the results as appropriate according to the review of the data package.

Two facts should be noted by all data users. First, **the “R” qualifier means that the laboratory-reported value is unusable.** In other words, due to significant quality control problems, the analysis is invalid and provides no information as to whether the compound is present or not. Rejected values should not appear on data tables because they cannot be relied upon, even as a last resort. Second, **no analyte concentration is guaranteed to be accurate even if all associated quality control is acceptable.** Strict quality control conformance serves only to increase confidence in reported results; any analytical result will always contain some error.

The data user is also cautioned that the validation effort is based on the raw data printouts as provided by the laboratory. Software manipulation cannot be routinely detected during validation; unless otherwise stated in the report, these kinds of issues are outside the scope of this review.





## **I. Holding Times, Preservation and Sample Integrity**

The soil samples were collected on March 11, 2005. The Composite Sample was digested and analyzed well within acceptable holding times (28 days for mercury and 180 days for the remaining analytes).

The chain of custody record indicated that no ice was present in the sample cooler and no temperature of the sample cooler was reported by the laboratory on the chain of custody record or on the Cooler Receipt Log. As elevated temperatures would not be expected to have an adverse effect on these analytes, no data were qualified on this basis.

The Chain of Custody Record and Cooler Receipt Log also indicated that the samples for compositing were received intact in Ziploc bags but no custody seals were used on the sample cooler.

A copy of the FedEx airbill was provided by the laboratory on April 4, 2005, to document custody of the samples between the field and the laboratory.

## **II. Mass Spectrometer Tune**

*No analytes were determined by ICP-MS.*

## **III. Calibrations**

Inductively coupled plasma-atomic emission spectroscopy (ICP-AES) analyses were performed on a trace ICP-AES instrument identified as "TJA61E Trace ICP 3." The samples reported in this data set were analyzed in two analysis series on March 22-23 and 25, 2005.

Mercury analyses were performed in a single analysis series run on March 24, 2005 on a cold vapor atomic absorption (CVAA) system identified as "PE CVAA 1."

Initial calibration verification (ICV) standards were performed for all of the target analytes after each initial calibration. All ICV percent recoveries (%R) were correctly calculated, accurately reported, and within the validation guideline-specified control limits (90-110% for analytes determined by ICP-AES and 80-120% for mercury).

Continuing calibration verification (CCV) standards were run at the appropriate frequency (after every ten samples) in all of the analysis series. All %Rs were correctly calculated, accurately reported, and within the validation guideline-specified control limits (90-110% for analytes determined by ICP-AES and 80-120% for mercury).



A linearity check for mercury was performed at the start of the analysis series and gave an acceptable correlation coefficient ( $>0.995$ ).

The analysis of “CRI” standards in the ICP-AES analyses and “CRA” standards in the mercury analyses are not required by SW-846 methods and were not performed in association with the reported analyses.

#### **IV. Blanks**

Initial and continuing calibration blanks (ICBs and CCBs) were analyzed at the appropriate frequencies throughout each analytical sequence. No target analytes were detected in any of the ICBs or CCBs.

Aluminum (15.0695 mg/kg), barium (0.140 mg/kg), beryllium (-0.066 mg/kg), calcium (20.179 mg/kg), chromium (0.094 mg/kg), copper (0.063 mg/kg), lead (0.495 mg/kg), magnesium (6.370 mg/kg), manganese (0.225 mg/kg), potassium (10.530 mg/kg), sodium (18.522 mg/kg), vanadium (0.0573 mg/kg), iron (9.882 mg/kg), and zinc (9.053 mg/kg) were detected in the solid-matrix preparation blanks (PBs). With regard to PBs, the validation guidance document indicates that if an analyte is detected in the PB below the contract required quantitation limit (CRQL), “no correction of the sample results should be performed.” For these samples, the estimated quantitation limit (EQL) was substituted for the CRQL. Only aluminum, lead, and manganese were detected above the EQLs and beryllium was detected below the negative EQL. Based on a sample concentration less than five times the absolute value of the negative result reported in the associated PB, the result for beryllium in Composite Sample was qualified as J-. Results for aluminum, lead, manganese, and the remaining target analytes noted above were significantly greater than the concentrations detected in the PB (i.e., all were greater than five times the concentration detected in the PB); therefore, results for these analytes were not qualified on this basis.

No field-submitted blanks were included in this data set.

#### **V. ICP Interference Check Sample**

ICP interference check sample analyses were performed at the appropriate frequency throughout all analysis series. Target analyte recoveries were acceptable (80-120%) in all cases.

## VI Spiked Sample Analyses

### A. Laboratory Control Samples

A solid-matrix laboratory control sample pair (LCS/LCSD) was prepared and analyzed with each analysis series. Each LCS/LCSD pair included all of the target analytes required for that analytical sequence. All percent recovery (%R) and relative percent difference (RPD) values were correctly calculated, accurately reported, and within the analyte-specific acceptance limits specified by the laboratory on the summary forms.

### B. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analyses

Composite Sample was prepared as an MS/MSD pair for all of the target analytes. All %R and RPD values were correctly calculated and accurately reported. For the target analytes with an amount of spike added greater than four times the concentration of the analyte in the original analysis of Composite Sample, %Rs and RPDs were within the analyte-specific acceptance limits specified by the laboratory on the summary forms with the following exception:

Analyte	MS	MSD	Acceptance Limits
antimony	6.2%	8.3%	80-120%

Based on excessively low recoveries (<10%) in both of the matrix spike analyses, the result for antimony in Composite Sample was rejected (R) based on professional judgment.

For the target analytes with an amount of spike added less than four times the concentration of the analyte detected in the original analysis of Composite Sample, acceptable recoveries would not be expected. Since the amount of spike added is insignificant, this presents an opportunity to compare precision among the triplicate analyses (sample, MS, and MSD). Based on professional judgment, an acceptance limit of  $\leq 25\%$  RSD was used to evaluate the precision among results reported for aluminum, barium, copper, iron, lead, manganese, and zinc. Acceptable precision among the triplicate results was observed (1.3% to 10.4%) in all cases.

Post digestion spike recoveries for all of the TAL target analytes in Composite Sample were acceptable (75-125% acceptance limits).

## VII. Duplicate Sample Analyses

### A. Laboratory Duplicate Analysis

No laboratory duplicate analysis was performed on a sample in this data set.

#### B. Field Duplicate Analyses

No field duplicate pair was identified in this sample set.

### VIII Internal Standard Performance

The sample in this data set was not analyzed by ICP-MS; therefore, this section is not applicable to this data set.

### IX ICP Serial Dilution

ICP-AES serial dilution analysis were performed on Composite Sample. Results for elements with initial (undiluted) results greater than 10 times the instrument detection limit for analytes determined by ICP-AES were acceptable (<10% Difference) in all cases.

### X. Sample Results Verification

All sample results were correctly calculated and accurately reported from the raw data, including adjustments for the solid-matrix preparation procedures, dilutions, and percent solids.

Instrument detection limits (IDLs) were reported for both instruments used to perform the reported sample results. IDLs were established on January 5, 2004, on the ICP-AES instrument and on July 5, 2004, on the instrument used to perform the mercury analyses. The analytical methods specify that IDLs must be established within three months of the associated sample analyses.

Linear ranges were established on January 27, 2005, on the instrument used to perform the ICP-AES analyses. Method 6010B specifies that linear ranges must be established within three months of the associated sample analyses.

Interelement correction factors were performed on December 31, 2003, on the instrument used to perform ICP-AES analyses. Method 6010B specifies that interelement correction factors must be established within one year of the associated sample analyses.

Based on professional judgment, as concentrations approach the IDL the accuracy of the measurement decreases; values closer to the EQL, however, are probably quite accurate. Therefore, a guideline of two times the IDL was used to determine whether the reported results warranted qualification. Sample results below the respective reporting limit, less than two times the IDL, and not otherwise qualified were qualified as estimated (J). No sample results met this criteria and no data were qualified on this basis.

## **XI. Documentation**

Chain of custody records were present and accurately completed for all samples reported in this data package with the following exceptions:

- The samples used to make the Composite Sample were collected on March 11, 2005, relinquished on March 18, 2005, and received by the laboratory on March 21, 2005. The custody and physical preservation of these samples during these time gaps were not disclosed in the data package.
- The section of the chain of custody record for recording the temperature and condition of the samples on laboratory receipt were not completed on either document. Although this information was provided on the Cooler Receipt Log, this information should also be recorded on each chain of custody record for all future sampling efforts.
- The laboratory did not include the year or the time of laboratory receipt on the second chain of custody record. For all future sampling efforts, the laboratory should be instructed to properly complete all appropriate sections of each chain of custody record.
- Improper editing was noted on one of the two chain of custody records. When necessary, corrections must be made by drawing a single line through the error, entering the correct information, and initialing and dating the correction.
- The chain of custody record and laboratory cooler receipt log indicate that no custody seals were used on the sample cooler.

No data presentation issues were noted in the data package received for review.

These chain of custody issues do not directly affect the validity of the reported results, but they could be problematic if these data are used in litigation.

## **XII. Overall Assessment**

Findings of the validation effort resulted in the following qualifications of sample results:

- Based on a sample concentration less than five times the absolute value of the negative result reported in the associated PB, the result for beryllium in Composite Sample was qualified as J-. The reported concentration may be biased low.



- Based on excessively low recoveries (<10%) in both of the matrix spike analyses, the result for antimony in Composite Sample was rejected (R) based on professional judgment.

The validator removed all “B,” “C,” “N,” and “\*” qualifiers applied by the laboratory.

Chain of custody documentation issues are discussed in Section XI.

This validation report should be considered part of the data package for all future distributions of the inorganics data.



**ATTACHMENT A**

**DATA SUMMARY FORM**

**Target Analyte List Metals in Soil Samples  
Sample Delivery Group Number 857319**

DATA SUMMARY FORM: INORGANICS  
SOIL SAMPLES  
(mg/kg)

Site Name: Eagle Zinc

Sampling Date: March 11, 2005

Laboratory Sample Delivery Group Number: 857319

Trillium Project No.: 05906

[illegible]





**ATTACHMENT B**

**LABORATORY ANSWER FORMS**

**Target Analyte List Metals in Soil Samples  
Sample Delivery Group Number 857319**

# En Chem

## Analytical Report Number: 857319

1241 Bellevue Street  
Green Bay, WI 54302  
920-469-2436

A Division of Pace Analytical Services, Inc.

Client : ENVIRON INTERNATIONAL CORP - DRFLD

Project Name : EAGLE ZINC

Project Number :

Field ID : COMPOSITE SAMPLE

Matrix Type : SOIL

Collection Date : 03/11/05

Report Date : 03/31/05

Lab Sample Number : 857319-001

### INORGANICS

Test	Result	EQL	Dilution	Units	Code	Anl Date	Prep Method	Anl Method
Aluminum	12000	2000	100	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Antimony	<del>15</del> R	15	10	mg/Kg as is	N*C	03/23/05	SW846 3050B	SW846 6010B
Arsenic	55	15	10	mg/Kg as is		03/25/05	SW846 3050B	SW846 6010B
Barium	220	2.0	10	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Beryllium	1.1 J- <del>5</del>	3.0	10	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Cadmium	22	3.0	10	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Calcium	5600	100	10	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Chromium	50	3.0	10	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Cobalt	630	5.0	10	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Copper	3700	20	10	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Iron	82000	10000	1000	mg/Kg as is		03/25/05	SW846 3050B	SW846 6010B
Lead	7100	100	100	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Magnesium	3200	50	10	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Manganese	2500	2.0	10	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Mercury	0.43	0.040	1	mg/kg as is		03/24/05	SW846 7471A	SW846 7471A
Nickel	1600	10	10	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Potassium	660	500	10	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Selenium	< 15	15	10	mg/Kg as is	C	03/23/05	SW846 3050B	SW846 6010B
Silver	58	5.0	10	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Sodium	1600	1000	10	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Thallium	8.4 <del>5</del>	20	10	mg/Kg as is		03/25/05	SW846 3050B	SW846 6010B
Vanadium	34	5.0	10	mg/Kg as is		03/23/05	SW846 3050B	SW846 6010B
Zinc	180000	2000	1000	mg/Kg as is		03/25/05	SW846 3050B	SW846 6010B

see 04/05/05